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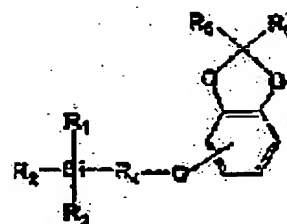
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(54) ORGANIC SILICON COMPOUND HAVING PROTECTED CATECHOL GROUP AND METHOD FOR PRODUCING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an alkoxy silane having catechol group protected with an organic substituent and useful for new synthetic process such as organic synthesis, production of new resin, method for surface-treatment of materials, modification of resins, etc.

SOLUTION: The organic silicon compound having a protected catechol group is expressed by general formula 1 (R1 to R3 are each independently a 1-6C alkyl, phenyl or an alkoxy provided that at least one of R1 to R3 is an alkoxy; R5 and R6 are each independently a 1-6C alkyl or phenyl; and R4 is a 2-6C alkylene).



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LEGAL STATUS

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DETAILED DESCRIPTION

[Detailed Description of the Invention]**[0001]**

[Field of the Invention] While excelling in the feel which the triglycerol denaturation silicone compound which has the specific chemical structure was used [feel] for this invention as a surfactant, and made the salt live together, emulsion stability is good and it is related with the cosmetics which can form the makeup film excellent in water repellence and endurance. Moreover, by blending with a cleaning agent, dirt omission is related with the cosmetics which the good after [a top] skin carries out gently and which were excellent in feel.

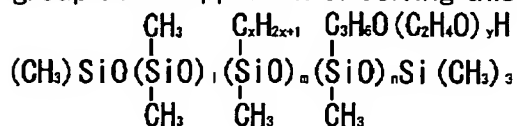
[0002]

[Description of the Prior Art] Generally, the sebum secreted from the skin especially in a makeup by the oils with which cosmetics are blended although the secrete from a man causes messy makeup, such as sweat, a tear, and sebum, is added, and it has become the big factor of messy makeup to wet the fine particles of cosmetics superfluously. Then, in order to reduce the oils in the cosmetics which remain on the skin, volatile oil agents, such as octamethylcyclotetrasiloxane and decamethyl cyclopentasiloxane, are used as some oils blended. Moreover, friction, water, etc. become the external factor which worsens makeup ****. Then, preventing losing water-soluble materials, sebum, etc. in the skin, in order to improve the badness of makeup **** which happens with water-soluble matter, such as sweat and a tear, blending silicon oil the making the protective effect of the skin maintain purpose, and making water repellence high is performed. For example, since it has the descriptions, such as a light feel and outstanding water-repellent and high safety, the silicon oil represented by dimethylpolysiloxane is used abundantly as oils used for cosmetics in recent years. Thus, although polysiloxanes have the property which was excellent as cosmetics oils, the concordance to the skin is bad, the feels carried out gently run short and there is a field inadequate in feel -- there is a feeling of jarring.

[0003] In recent years, in the water-in-oil type emulsification constituent (W/O emulsification object), silicon oil is used as oils. However, the water-in-oil type emulsification object containing this silicon oil is difficult to obtain the good emulsification object of stability in emulsifiers, such as a polyoxyalkylene fatty-acid-ester system used conventionally. Then, the approach of using polyoxyalkylene denaturation organopolysiloxane with sufficient silicon oil and compatibility (polyether denaturation silicone) as a surfactant is proposed to the above-mentioned water-in-oil type emulsification object (for example, refer to JP, 61-293903, A, 61-293904, 62-187406, 62-215510, and No. 216635 [62 to] each of this official report).

[0004] However, although ester oil and a hydrocarbon oil were used together with silicon oil as oils in many cases when the emulsification object aiming at a cosmetics application was

obtained, the above-mentioned polyether denaturation silicone was inferior to the emulsification force in such a mixed oils system, and had the fault that it was difficult to obtain a stable water-in-oil type emulsification object. The approach using the organopolysiloxane which had the long-chain alkyl group expressed with the following type and the polyoxyalkylene group as an approach of solving this fault as an emulsifier is proposed by JP,61-90732,A.



However, although the emulsification force was excellent in the mixed oils system with many ester oil and hydrocarbon oils, the above-mentioned organopolysiloxane compound had the fault that it was common to be stable and for it to be difficult to obtain an emulsification object without aging, when it was a mixed oils system with much silicon oil. Therefore, it has the emulsifiability ability which was excellent to the oils used for general cosmetics, such as silicon oil, ester oil, and a hydrocarbon oil, and a suitable emulsifier for the cosmetics application which can secure stability with the passage of time was desired.

[0005] On the other hand, in the field of a skin cleaning agent, since makeup cosmetics, such as a lip stick, foundation, eye shadow, an eyeliner, and mascara, contain solid oil so much, they are difficult to drop makeup dirt to the cleaning agent using usual soap completely, since the solubilization ability or emulsification ability to these oil are not enough. So, in such a case, the cleaning agent which made the oily basis the subject was used. However, makeup is hard to come off recently, the good makeup cosmetics of makeup rice cake are developed, and Kamiichi of the makeup cosmetics with which the polymer with high oils and film forming ability, such as annular silicone, especially with many sweat rates was blended is carried out as a ** summer. Moreover, also in hair cosmetics, protect hair or Hari and chewiness are given to hair, or various matter, such as a polymer with high high-polymer silicone and film forming ability, is blended in order to take out admiration smoothly. Thus, makeup was hard to come off, and although the cleaning agent using a nonionic surfactant and polyether denaturation silicone as the washing approach of cosmetics excellent in the protective effect of hair etc. had so far been used, development of a cleaning agent constituent with a more high cleaning effect was desired with the functional improvement in the film forming ability as cosmetics etc.

[0006] Various glycerol denaturation silicone is reported as effective denaturation silicone as an Nonion system surfactant, improving the badness of the concordance to a feeling of jarring and the skin peculiar to silicone. As oils, the silicone indicated by JP,6-157236,A and the 9-71504 official report and the silicone of the fluorine alkyl covariance indicated by the JP,10-310504,A - No. 310509 official report are specifically raised, and the cosmetics indicated by the glycerol denaturation silicone compound indicated by JP,62-34039,B, patent No. 2613124, patent No. 2844453, etc., JP,8-22811,B, patent No. 2587797, and patent No. 2601738 as a surfactant are mentioned. As a polyhydric-alcohol denaturation silicone compound which has a hydroxyl group, sugar and a polysaccharide denaturation silicone compound are reported further. The denaturation silicone compound which has sugar residue is indicated by JP,5-186596,A, and the application as an emulsifier is indicated by each official report of JP,6-145023,A, 7-41414, and 7-41416. When anything uses polyhydric-alcohol denaturation silicone compounds, such as a glycerol, while decreasing a feeling of jarring peculiar to silicone, it is used for the purpose which was described when using the oils of a glycerol system and which sticks and controls admiration by addition of silicone.

[0007]

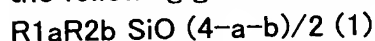
[Problem(s) to be Solved by the Invention] this invention person etc. thickens by mixing with water like polyether denaturation silicone, or It not only does not discover a feeling of stickiness, but the prompt detergency over oil dirt etc. is shown. The compatibility in the inside of the feeling of stickiness which was the fault of conventional polyhydric-alcohol denaturation silicone, and silicone system oils is improved. There is water repellence at a light feel, and usability is good and has the feeling of use carried out gently. The result wholeheartedly studied about the denaturation silicon oil agent with the emulsifiability ability and emulsion stability which were excellent in each oils list, such as a denaturation silicon oil agent by which adhesion with the skin is impressed, and silicon oil used for cosmetics, to these mixed oils, A header and this invention were reached [that the cosmetics which made the triglycerol denaturation silicone compound and the salt contain are good, and]. Therefore, the 1st purpose of this invention has the emulsification force and emulsion stability which were excellent while excelling in the feel, and is to offer the cosmetics which the back skin carries out gently. The 2nd purpose of this invention has prompt detergency to oil dirt etc., and is to offer a cleaning agent constituent with the outstanding feeling of use.

[0008]

[Means for Solving the Problem] The above-mentioned purpose of this invention used as A component the triglycerol denaturation silicone compound expressed with $R_1aR_2bSiO(4-a-b)/2$, and was attained by the cosmetics which contain at least one sort of salts as a B component.

[0009]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail. The triglycerol denaturation silicone compound which is A component used in this invention is expressed with the following general formula (1).

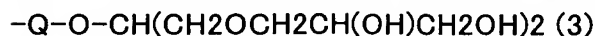


As an example of R_1 , fluorine permutation alkyl groups, such as aralkyl radical; triphloropropyl groups, such as aryl group; benzyls, such as cycloalkyl radical; phenyl groups, such as alkyl group; cyclopentyl groups, such as a methyl group, an ethyl group, a propyl group, butyl, a pentyl radical, a hexyl group, a heptyl radical, an octyl radical, a nonyl radical, a decyl group, a hexadecyl radical, and an octadecyl radical, and a cyclohexyl radical, and a tolyl group, and a phenethyl radical, and a heptadeca FURORO decyl group, etc. can be mentioned. Furthermore, carboxy permutation alkyl groups, such as amino permutation alkyl group; 3-carboxy propyl groups, such as 3-aminopropyl radical and 3-[(2-aminoethyl) amino] propyl group, etc. are mentioned.

[0010] A part of R_1 may be the organic radical expressed with general formula- $CdH_{2d-O-(C_2H_4O)_e(C_3H_6O)_fR_3}$. R_3 is an organic radical expressed with the monovalence hydrocarbon group of carbon numbers 4-30, or $R_4-(CO)-$, and R_4 is the monovalence hydrocarbon group of carbon numbers 1-30. Each is the integer of $0 \leq d \leq 15$, $0 \leq e \leq 50$, and $0 \leq f \leq 50$, and d, e, and f are alcohol residue and alkenyl ether addition product residue. For example, $d=0$ At the time : If it is $-O-(C_2H_4O)_e(C_3H_6O)_fR_3$ and $e=0$, and $f=0$, it is the alkoxy group of carbon numbers 4-30. As the example For example, cetyl alcohol from lower alkoxy groups, such as a butoxy radical, oleyl alcohol, me, such as stearyl alcohol, -- fatty-acid residue, such as high-class alkoxy groups, such as an yloxy radical and a steer ROKISHI radical, or an acetic acid, a lactic acid, butanoic acid, oleic acid, stearin acid, and a behenyl acid, is mentioned. If it is e and $f > 1$, it will become the alcohol residue of the alkylene oxide addition product (an end is a hydroxyl group) of higher alcohol. Especially when d is 1 or more, $e=0$, and $f=0$, it is

desirable that d is 3, 5, or 11, and it serves as the allyl compound ether, the pentenyl ether, and undecenyl ether residue in this case. By the substituent of R3, it becomes for example, allyl compound stearyl ether residue, pentenyl behenyl ether residue, or undecenyl oleyl ether residue. When e or f is not 0, an alkoxy group and an ester group will exist through polyoxyalkylene. Here, when it is e and d is 0 whatever the f, it is desirable that d is 3-5 since **** is strong as it may be inferior to hydrolysis-proof nature and d is 15 or more. Moreover, it is desirable that 50% or more of the R1 whole is a methyl group, and it is desirable that especially 70% or more is a methyl group. A methyl group may be 100%.

[0011] R2 is expressed with the following general formula (3).

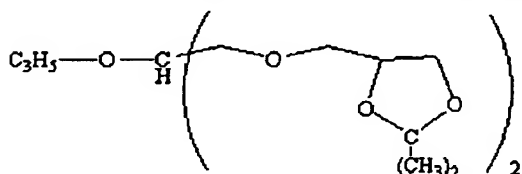


Q is the bivalence hydrocarbon group of the carbon numbers 3-20 which may contain ether linkage and an ester bond among an upper type. For example, $-(CH_2)_2-$, $-(CH_2)_3-$, $-CH_2CH(CH_3)CH_2-$, $-CH_2CH_2-$, $-(CH_2)_5-$, $-(CH_2)_6-$, $-(CH_2)_7-$, $-CH_2CH_2CH_2-$, $-(CH_2)_2-CH(CH_2CH_2CH_3)-$, $-CH_2-CH(CH_2CH_3)-$, $-(CH_2)_3-O-(CH_2)_2-$, $-(CH_2)_3-O-(CH_2)_2-O-(CH_2)_2-$, $-(CH_2)_3-O-CH_2CH(CH_3)-$, $-CH_2-CH(CH_3)-COO(CH_2)_2-$, etc. can be illustrated.

[0012] The above-mentioned silicone compound of a formula (1) used by this invention can compound easily the allyl compound ether compound expressed in following (ii) as the ORGANO hydrogen polysiloxane, the allyl compound ether compound which protected the alcohol expressed especially below (iii) preferably, and the allyl compound ether compound further expressed with alkylene compounds, such as a hexene, or following (i) depending on the case by carrying out an addition reaction to the bottom of existence of a platinum catalyst or a rhodium catalyst.

$C_3H_5-O-(C_2H_4O)_e(C_3H_6O)_f-R_3$ (i) (however, R3, e, and f in a formula are the same as the case of Rabove mentioned 1 respectively.)

$C_3H_5-O-CH(CH_2OCH_2CH(OH)CH_2OH)_2$ (ii)



(iii)

Here, as an ORGANO hydrogen polysiloxane, although the shape of a straight chain and annular any are sufficient, in order to advance an addition reaction smoothly, it is desirable that it is mainly a straight chain-like. the mixed rate of the amount of the ORGANO hydrogen polysiloxane used, and the total quantity of the organic compound expressed with the above-mentioned general formula (ii) or (iii) the allyl compound ether compound expressed, an alkylene compound, and/or the above-mentioned general formula (i) -- the mole ratio of a SiH radical and an end partial saturation radical -- 0.5-2.0 -- it is 0.8-1.2 preferably.

[0013] As for the above-mentioned addition reaction, it is desirable to carry out under existence of a platinum catalyst or a rhodium catalyst, and the catalyst of a chloroplatinic acid, alcoholic denaturation chloroplatinic acid, and chloroplatinic acid-vinyl siloxane complex etc. is specifically used suitably. In addition, although the amount of the catalyst used can be made into the amount of catalysts, it is desirable that it is 20 ppm or less that it is 50 ppm or less in platinum or the amount of rhodiums especially preferably. The above-mentioned addition

reaction may be performed in an organic solvent if needed. As an organic solvent, halogenated hydrocarbon, such as aliphatic series, such as aromatic hydrocarbon, such as fatty alcohol, such as a methanol, ethanol, 2-propanol, and a butanol, toluene, and a xylene, n pentane, n-hexane, and a cyclohexane, or alicyclic hydrocarbon, dichloromethane, chloroform, and a carbon tetrachloride, etc. is mentioned, for example. Although especially addition reaction conditions are not limited, it is desirable to make it react under reflux for 1 to 10 hours. To compound especially using an allyl compound ether compound (iii), it is necessary to perform the deacetone reaction after an addition reaction. A reaction adds 10 - 30% of 0.1 M-HCl water to a reaction mixture, and is attained by carrying out heating churning at the temperature of 50-100 degrees C for 5 to 10 hours.

[0014] a -- 1.0-2.5 -- it is 1.2-2.3 preferably. When a is smaller than 1.0, it is inferior to compatibility with oils, and difficult to get in a stable emulsification object. Since it will become lacking in a hydrophilic property if a is larger than 2.5, it is difficult to get in a too stable emulsification object. b -- 0.001-1.5 -- it is 0.05-1.0 preferably. If b is smaller than 0.001, since it will become lacking in a hydrophilic property, it is difficult to get in a stable emulsification object, and since a hydrophilic property will become high too much if larger than 1.5, it is difficult to get in a too stable emulsification object. Although especially the weight average molecular weight of the silicone compound expressed with the aforementioned formula (1) is not limited when using it for an emulsification object, it is desirable 500-200,000, and that it is especially the molecular weight of 1,000-100,000. Although it is desirable that it is 4,000 or less as for the weight average molecular weight of the silicone compound expressed with the aforementioned formula (1) on the other hand when using it for a skin washing constituent, it is desirable that it is 2,000 especially or less, and it is most desirable that it is 1,500 or less.

[0015] A salt is used for the cosmetics of this invention as a B component with the triglycerol denaturation silicone of A component. As this salt, mineral salt, an organic-acid salt, an amine salt, and an amino acid salt are mentioned. As mineral salt, for example The sodium salt of inorganic acids, such as a hydrochloric acid, a sulfuric acid, carbonic acid, and a nitric acid, Potassium salt, magnesium salt, a calcium salt, an aluminum salt, a zirconium salt, zinc salt, etc.; as an organic-acid salt For example, the salt of organic acids, such as an acetic acid, a dehydroacetic acid, a citric acid, an apple acid, a succinic acid, an ascorbic acid, and stearin acid; as an amine salt and an amino acid salt, there is a salt of amino acid, such as a salt of amines, such as triethanolamine, and glutamic acid, etc., for example. Moreover, in addition to this, the neutralization salt [complex / salts, such as hyaluronic acid and chondroitin sulfate, / aluminum zirconium glycine] of the acid-alkali further used in a cosmetics formula etc. can be used.

[0016] According to the purpose, one sort or two sorts or more of oils can be used for the cosmetics of this invention as a C component. As such oils, if used for the usual cosmetics, anything of a solid-state, a semisolid, and liquefied oils can be used. for example, as natural-animal-and-plant-oil fat and semisynthesis fats and oils An avocado oil, the linseed oil, an almond oil, IBOTAROU, perilla oil, olive oil, Cacao butter, a kapok low, kaya oil, a carnauba wax, liver oil, a candelilla low, Beef tallow, neat's foot lipid, beef bone fat, hardening beef tallow, an apricot kernel oil, spermaceti wax, hardened oil, a wheat germ oil, sesame oil, and rice -- germ oil, rice bran oil, a sugarcane low, and a sasanqua oil -- Safflower oil, shea butter, a SHINAGIRI oil, a cinnamon oil, jojoba wax, Squalene, a shellac low, a turtle oil, soybean oil, tea seed oil, camellia oil, Oenotherae Biennis oil, corn oil, lard, rapeseed oil, Japanese tung oil, bran wax, Germ oil, horse fat, a par chic oil, palm oil, palm kernel oil, castor oil, hydrogenated castor

oil, Castor oil fatty-acid methyl ester, sunflower oil, a grape oil, a bay berry low, Jojoba oil, a macadamia-nuts oil, yellow bees wax, a mink oil, cotton seed oil, a cotton low, Japan wax, Japan wax kernel oil, montan wax, palm oil, hardening palm oil, Tori palm-oil-fatty-acid glyceride, Mutton tallow, peanut oil, lanolin, liquefied lanolin, and reduction lanolin, lanolin alcohol, Hard lanolin, acetic-acid lanolin, lanolin fatty-acid isopropyl, the POE lanolin alcoholic ether, POE lanolin alcoholic acetate, a lanolin fatty-acid polyethylene glycol, the POE hydrogenation lanolin alcoholic ether, a yolk oil, etc. are mentioned. However, POE means a polyoxyethylene.

[0017] As a hydrocarbon oil, an ozokerite, squalane, squalene, a ceresin, Paraffin, paraffin wax, polyethylene wax, a polyethylene PORIPIRO pyrene wax, A liquid paraffin, pristane, a polyisobutylene, a micro crystallin wax, vaseline, etc.; as a higher fatty acid A lauric acid, a myristic acid, a palmitic acid, stearin acid, behenic acid, Undecylenic acid, oleic acid, linolic acid, a linolenic acid, an arachidonic acid, eicosapentaenoic acid (EPA), docosa-hexaenoic acid (DHA), isostearic acid, 12-hydroxy stearin acid, etc. are mentioned.

[0018] As a higher-alcohol oil, lauryl alcohol, myristyl alcohol, palmityl alcohol, stearyl alcohol, behenyl alcohol, hexadecyl alcohol, oleyl alcohol, isostearyl alcohol, a hexyl dodecanol, an octyl dodecanol, the cetostearyl alcohol, 2-DESHIRUTETORADESHINORU, cholesterol, a phytosterol, the POE cholesterol ether, the mono-stearyl glycerol ether (batyl alcohol), mono-oleyl glyceryl ether (selachyl alcohol), etc. are mentioned.

[0019] As ester oil, diisobutyl adipate, adipic-acid 2-hexyl DESHIRU, Diheptylundecyl adipate, iso nonoic acid iso nonyl, A mono-isostearic acid N-alkyl glycol, isostearic acid isocetyl, Tori isostearic acid trimethylol propane, G 2-ethylhexanoic acid ethylene glycol, 2-ethylhexanoic acid cetyl, tree 2-ethylhexanoic acid trimethylol propane, Tetra--2-ethylhexanoic acid pentaerythritol, octanoic-acid cetyl, Octyldodecyl gum ester, oleic acid oleyl, oleic acid octyldodecyl, Oleic acid DESHIRU, neopentylglycol dicaprato, citric-acid triethyl, Succinic-acid 2-ethylhexyl, amyl acetate, ethyl acetate, butyl acetate, Stearin acid isocetyl, butyl stearate, diisopropyl sebacate, Di-2-ethylhexyl sebacate, lactic-acid cetyl, lactic-acid Millis Chill, Palmitic-acid isopropyl, palmitic-acid 2-ethylhexyl, palmitic-acid 2-hexyl DESHIRU, Palmitic-acid 2-heptyl undecyl, 12-hydroxy stearyl acid cholesteryl, Dipentaerythritol fatty acid ester, myristic-acid isopropyl, Myristic-acid octyldodecyl, myristic-acid 2-hexyl DESHIRU, Myristic-acid Millis Chill, dimethyl octanoic-acid hexyl DESHIRU, lauric-acid ethyl, Lauric-acid hexyl, N-lauroyl-L-glutamic acid-2-octyldodecyl ester, malate diisostearyl, etc.; as a glyceride oil The aceto glyceryl, TORIISO octanoic-acid glyceryl, Tori isostearic acid glyceryl, The Tori Isopar Rumi Ching acid glyceryl, monostearin acid glyceryl, G 2-heptyl undecanoic acid glyceryl, trimyristin acid glyceryl, myristic-acid isostearic acid diglyceryl, etc. are mentioned.

[0020] As silicon oil, dimethylpolysiloxane, a methylphenyl polysiloxane, Methil hydrogen polysiloxane, a dimethylsiloxane methylphenyl siloxane copolymer, etc., The straight chain of hypoviscosity to hyperviscosity or the organopolysiloxane of the letter of branching, octamethylcyclotetrasiloxane, Decamethyl cyclopentasiloxane, a dodeca methyl cyclohexa siloxane, Cyclosiloxanes, such as tetramethyl tetrahydrogen cyclotetrasiloxane and tetramethyl tetra-phenyl cyclotetrasiloxane, Silicone rubber, such as a dimethylsiloxane methylphenyl siloxane copolymer of the shape of high-polymer gum-like dimethylpolysiloxane and gum, And the cyclosiloxane solution of silicone rubber, a trimethylsiloxo silicic acid, High-class alkoxy denaturation silicone, such as a cyclosiloxane solution of a trimethylsiloxo silicic acid, and steer ROKISHI silicone, The melt of higher-fatty-acid denaturation silicone, alkyl denaturation silicone, amino denaturation silicone, fluorine denaturation silicone, silicone resin, and silicone resin etc. is mentioned. As fluorine system oils, a perfluoro polyether, a perfluoro decalin, a

perfluorooctane, etc. are mentioned. Although the loadings of the oils as these C components change also with ** systems, 1 - 98% of the weight of the range of the whole cosmetics is suitable for them.

[0021] According to the purpose, the compound which has an alcoholic hydroxyl group in one sort or two sorts or more of molecular structures can be used for the cosmetics of this invention as a D component. as the compound which has the above-mentioned alcoholic hydroxyl group -- sugar-alcohol, such as lower alcohol, such as ethanol and isopropanol, a sorbitol, and a maltose, etc. -- it is -- sterols, such as cholesterol, a sitosterol, a phytosterol, and lanosterol, a butylene glycol, propylene glycol, and a jib -- there is polyhydric alcohol, such as a CHIREN glycol, etc. 0.1 - 98% of the weight of the range of the whole cosmetics is suitable for loadings.

[0022] According to the purpose, a water soluble polymer or a water bloating tendency macromolecule can also be used for the cosmetics of this invention as one sort or two sorts or more, and an E component. As the above-mentioned water soluble polymer, for example Gum arabic, tragacanth, Galactan, carob gum, guar gum, karaya gum, a carrageenan, pectin, an agar, KUINSU seed (quince), and starch (rice and corn --) Vegetable system macromolecules, such as a potato, wheat, ARUGE colloid, tolan TOGAMU, and locust bean gum; Xanthan gum, Microorganism system macromolecules, such as a dextran, SAKUSHINO glucan, and a pullulan; A collagen, Animal system giant molecules, such as casein, albumin, and gelatin; Carboxymethyl starch, Starch system giant molecules, such as methyl hydroxypropyl starch; Methyl cellulose, Ethyl cellulose, methyl hydroxypropylcellulose, a carboxymethyl cellulose, A hydroxymethyl cellulose, hydroxypropylcellulose, a nitrocellulose, A cellulose sodium sulfate, carboxymethylcellulose sodium, Cellulose system macromolecules, such as crystalline cellulose and cellulose powder; Sodium alginate, Alginic-acid system giant molecules, such as propylene glycol alginate; Polyvinyl methyl ether, vinyl system giant-molecules [, such as a carboxyvinyl polymer,]; -- polyoxyethylene system giant-molecule; -- polyoxyethylene polyoxypropylene copolymer system giant-molecule; -- sodium polyacrylate -- Acrylic giant molecules, such as polyethylacrylate and polyacrylamide; Polyethyleneimine, Other synthetic water soluble polymers, such as a cation polymer; there are inorganic system water soluble polymers, such as a bentonite, the magnesium aluminum silicate, a montmorillonite, beidellite, nontronite, saponite, hectorite, and a silicic acid anhydride, etc. Moreover, coat formation agents, such as polyvinyl alcohol and a polyvinyl pyrrolidone, are also contained in these water soluble polymers. As loadings, 0.01 - 25% of the weight of the range of the whole cosmetics is suitable.

[0023] According to the purpose, water can also be blended with the cosmetics of this invention as an F component. Although the loadings change also with ** systems, 1 - 99% of the weight of the range of the whole cosmetics is suitable for them.

[0024] According to the purpose, fine particles can be further used for the cosmetics of this invention as a G component. As the above-mentioned fine particles, if used for the usual cosmetics, the configurations (globular shape, needle, tabular, etc.) or particle diameter (the shape of haze, a particle, pigment class, etc.), and particulate structures (porosity, quality of nonporous, etc.) cannot be asked, but anything can be used. As such a powder object, inorganic fine particles, organic fine particles, surfactant metal salt fine particles, a colored pigment, a pearl pigment, a metal powder pigment, natural coloring matter, etc. are mentioned, for example.

[0025] As an example of inorganic fine particles, titanium oxide, a zirconium dioxide, a zinc oxide, Cerium oxide, magnesium oxide, a barium sulfate, a calcium sulfate, Magnesium sulfate, a calcium carbonate, a magnesium carbonate, talc, a mica, A kaolin, a sericite, a muscovite,

synthetic mica, phlogopite, lepidolite, a biotite, A lithia mica, a silicic acid, a silicic acid anhydride, an aluminum silicate, a magnesium silicate, The magnesium aluminum silicate, a calcium silicate, silicic-acid barium, Silicic-acid strontium, a tungstic-acid metal salt, hydroxyapatite, A vermiculite, a HAIJI light, a bentonite, a montmorillonite, hectorite, a zeolite, ceramic powder, calcium diphosphate, an alumina, an aluminum hydroxide, boron nitride, boron nitride, a silica, etc. are mentioned.

[0026] As an example of organic fine particles, polyamide powder, polyester powder, Polyethylene powder, polypropylene powder, polystyrene powder, Polyurethane powder, benzoguanamine powder, the poly methyl benzoguanamine powder, Tetrafluoroethylene powder, polymethylmethacrylate powder, Nylon powder, such as cellulose powder, silk powder, 12 nylon, and 6 nylon, Bridge formation mold silicone impalpable powder with the structure which constructed the bridge in dimethyl silicone, The impalpable powder of poly methyl silsesquioxane, a styrene acrylic-acid copolymer, A lauroyl lysine etc. is mentioned in a divinylbenzene styrene copolymer, vinyl resin, a urea-resin, phenol resin, a fluororesin, silicone resin, acrylic resin, melamine resin, an epoxy resin, polycarbonate resin, microcrystal fiber fine particles, and the end of starch.

[0027] As surfactant metal salt fine particles (metallic soap), zinc stearate, aluminum stearate, calcium stearate, magnesium stearate, myristic-acid zinc, myristic-acid magnesium, cetyl phosphoric-acid zinc, cetyl calcium phosphate, cetyl phosphoric-acid zinc sodium, etc. are mentioned. As an example of a colored pigment, the inorganic red system pigment of ferrous oxide, an iron hydroxide, and titanitic-acid iron, Inorganic yellow system pigments, such as inorganic brown system pigments, such as gamma-iron oxide, yellow oxide of iron, and ocher, Inorganic black system pigments, such as black oxide of iron and carbon black, manganese violet, Inorganic purple system pigments, such as cobalt violet, chromium hydroxide, chrome oxide, What lake-ized inorganic blue system pigments, such as inorganic green system pigments, such as cobalt oxide and titanitic-acid cobalt, Berlin blue, and ultramarine blue, and tar system coloring matter, the thing which lake-ized natural coloring matter, the synthetic-resin fine particles which compound-ized these fine particles are mentioned.

[0028] As a pearl pigment, a titanium oxide covering mica, a titanium oxide covering mica, Bismuth oxychloride, titanium oxide covering bismuth oxychloride, titanium oxide covering talc, a scales foil, a titanium oxide covering coloring mica, etc.; as a metal powder pigment Aluminum powder, kappa powder, stainless steel powder, etc.; as tar dye Red No. 3, red No. 104, red No. 106, red No. 201, red No. 202, Red No. 204, red No. 205, red No. 220, red No. 226, red No. 227, Red No. 228, red No. 230, red No. 401, red No. 505, yellow No. 4, Yellow No. 5, yellow No. 202, yellow No. 203, yellow No. 204, yellow No. 401, Blue No. 1, blue No. 2, blue No. 201, blue No. 404, green No. 3, green No. 201, Green No. 204, green No. 205, orange No. 201, orange No. 203, orange No. 204, orange No. 206, orange No. 207, etc.; as natural coloring matter, carminic acid, a laccainic acid, cull SAMIN, the brazilin, crocin, etc. are mentioned. Moreover, the fine particles which carry out absorption dispersion of the ultraviolet rays of particle titanium oxide, particle iron content titanium oxide, a particle zinc oxide, particle cerium oxide, those complex, etc. are also mentioned. These fine particles are the range which does not bar the effectiveness of this invention, and can use what was processed with silicon oil other than the triglycerol silicone compound which compound-izes or is expressed with common oils and said general formula (1), the fluorine compound, the surfactant, etc. moreover, these powder objects -- the need -- responding -- one sort -- or two or more sorts can be used.

[0029] Surfactants other than the triglycerol denaturation silicone compound which is A

component can also be used for the cosmetics of this invention as one sort or two sorts or more, and an H component. Anything can be used, if it is not restricted especially and used for the usual cosmetics as such a surface active agent, although there is an activator of anionic, cationicity, nonionic, and both sexes.

[0030] When it illustrates concretely below, as an anionic surfactant fatty-acid soap [, such as a sodium stearate and palmitic-acid triethanolamine,]; -- alkyl ether carboxylic-acid and its salt; -- the condensate salt of amino acid and a fatty acid -- An alkane sulfonate, an alkene sulfonate, the sulfonate of fatty acid ester, The sulfonate of a fatty-acid amide, a formalin condensed system sulfonate, an alkyl-sulfuric-acid ester salt, The second class fatty alcohol sulfate, alkyl, and an allyl compound ethereal sulfate ester salt, The sulfate salt of fatty acid ester, the sulfate salt of a fatty-acid ARUKI roll amide, Sulfate salts, such as Turkey red oil, alkyl phosphate, ether phosphate, Alkyl allyl compound ether phosphate, amide phosphate, a N-acylamino acid system activator, etc.; as a cationic surfactant Amine salts, such as an alkylamine salt, polyamine, and an amino alcohol fatty-acid derivative, alkyl quarternary ammonium salt, aromatic series quarternary ammonium salt, a pilus JIUMU salt, an imidazolium salt, etc. are mentioned.

[0031] As a nonionic surfactant, a sorbitan fatty acid ester, a glycerine fatty acid ester, Polyglyceryl fatty acid ester, propylene glycol fatty acid ester, Polyethylene glycol fatty acid ester, sucrose fatty acid ester, polyoxyethylene alkyl ether, Polyoxypropylene alkyl ether, polyoxyethylene alkyl phenyl ether, Polyoxyethylene fatty acid ester, polyoxyethylene sorbitan fatty acid ester, Polyoxyethylene sorbitol fatty acid ester, polyoxyethylene glycerine fatty acid ester, Polyoxyethylene propylene glycol fatty acid ester, polyoxyethylene castor oil, Polyoxyethylene hydrogenated castor oil, the polyoxyethylene phytostanol ether, The polyoxyethylene phytosterol ether, the polyoxyethylene cholestanol ether, The polyoxyethylene cholesteryl ether, a straight chain, or the polyoxyalkylene denaturation organopolysiloxane of the letter of branching, A straight chain or the polyoxyalkylene alkyl covariance organopolysiloxane of the letter of branching, an alkanol amide, the sugar ether, a sugar amide, etc.; a betaine, aminocarboxylate, an imidazoline derivative, etc. are mentioned as an amphoteric surface active agent. Moreover, as loadings, it is desirable that they are 0.1 – 20% of the weight of the whole cosmetics, and 0.2 – 10% of the weight of the range is especially suitable.

[0032] According to the purpose, one sort or two sorts or more of bridge formation mold organopolysiloxane can also be used for the cosmetics of this invention as an I component. As for this bridge formation mold organopolysiloxane, it is desirable that it is what is swollen to the hypoviscosity silicone of a 0.65mm²/second – 100.0mm²/second including this hypoviscosity silicone beyond a self-weight. Moreover, as for the cross linking agent of this bridge formation mold organopolysiloxane, it is desirable that it is what forms the structure of cross linkage by reacting between the hydrogen atoms which had two or more vinyl sexual response parts in the molecule, and were coupled directly with the silicon atom. Furthermore, this bridge formation mold organopolysiloxane may contain in a molecule a polyoxyalkylene part, an alkyl part, an alkenyl part, an aryl part, and at least one sort of parts chosen from the group which consists of a fluoro alkyl part. Although especially these bridge formation mold organopolysiloxane is not the objects limited, as a commercial item, there are KSG-15 made into gel with silicon oil, KSG-16, KSG-18, KSG-21 (all are the Shin-Etsu Chemical Co., Ltd. make), etc.

[0033] Moreover, as the above-mentioned bridge formation mold organopolysiloxane, what swells oils other than the hypoviscosity silicone of said 0.65mm²/second – a 100.0mm²/second in ***** beyond a self-weight is used. As for the cross linking agent of this bridge formation

mold organopolysiloxane, it is desirable that it is what forms the structure of cross linkage by reacting between the hydrogen atoms which had two or more vinyl sexual response parts in the molecule, and were coupled directly with the silicon atom. Furthermore, this bridge formation mold organopolysiloxane may contain in a molecule a polyoxyalkylene part, an alkyl part, an alkenyl part, an aryl part, and at least one sort of parts chosen from the group which consists of a fluoro alkyl part. Although especially these bridge formation mold organopolysiloxane is not the objects limited, as a commercial item, KSG-31 made into gel from the hydrocarbon oil or triglyceride oil, KSG-32, KSG-33, KSG-34, KSG-41, KSG-42, KSG-43, KSG-44 (all are the Shin-Etsu Chemical Co., Ltd. make), etc. are mentioned. As for the loadings of bridge formation mold organopolysiloxane, it is desirable that it is 0.01 – 40 % of the weight to the total amount of cosmetics, and it is especially desirable that it is 0.1 – 30 % of the weight.

[0034] According to the purpose, one sort or two sorts or more of silicone resin can also be used for the cosmetics of this invention as a J component. As for this silicone resin, it is desirable that they are an acrylic / silicone graft, or acrylic silicone resin of a block copolymer. Moreover, the acrylic silicone resin which contains in a molecule at least one sort of parts chosen from anion parts, such as a pyrrolidone part, a long-chain alkyl part, a polyoxyalkylene part and a fluoro alkyl part, and a carboxylic acid, can also be used. As for this silicone resin, it is desirable that it is the silicone reticulated compound expressed as MQ, MDQ, MT, MDT, and MDTQ as a constituent. This M, D, T, and Q show $R_3SiO_{0.5}$ unit, an R_2SiO unit, $RSiO_{1.5}$ unit, and SiO_2 unit, and, generally are used in the silicone industry, respectively. Generally silicone reticulated resin is known as MQ resin or MT, and MDT resin, and it may have the part indicated to be MDQ and MDTQ. It is specifically marketed as melts, such as octamethylcyclotetrasiloxane, and at least one sort of parts chosen from a pyrrolidone part, a long-chain alkyl part, a polyoxyalkylene part and a fluoro alkyl part, and an amino part may be contained in a molecule. When using silicone resin, such as acrylic silicone resin and a silicone reticulated compound, as for the loadings, it is desirable that it is 0.1 – 20 % of the weight to the total amount of cosmetics, and it is especially desirable that it is 1 – 10 % of the weight.

[0035] Furthermore, the component used for the usual cosmetics in the range which does not bar the effectiveness of this invention to the cosmetics of this invention, An oil solubility gelling agent, an organic denaturation clay mineral, resin, an antiperspirant, an ultraviolet ray absorbent, An ultraviolet absorption dispersion agent, a moisturizer, antiseptics, an antimicrobial agent, perfume, an antioxidant, pH regulator, A chelating agent, a refrigerant, an anti-inflammatory agent, the components for lustrous skin (a whitening agent, a cell activator, a surface deterioration improvement agent, a circulation accelerator, a skin astringent, antiseborrheic drug, etc.), vitamins, amino acid, a nucleic acid, hormone, a clathrate compound, the solidification agent for hair, etc. can be added.

[0036] As an oil solubility gelling agent, aluminum stearate, magnesium stearate, Metal soap, such as a zinc millimeter state; N-lauroyl-L-glutamic acid – Amino acid derivative; dextrin palmitic-acid ester, such as alpha and gamma-G n butylamine, Dextrin fatty acid ester, such as dextrin stearic acid ester and dextrin 2-ethylhexanoic acid palmitic-acid ester; Cane-sugar palmitic-acid ester, Sucrose fatty acid ester, such as cane-sugar stearic acid ester; A mono-benzylidene sorbitol, The gelling agent chosen from organic denaturation clay minerals, such as a benzylidene derivative of sorbitols, such as a JIBEN zylidene sorbitol, dimethylbenzyl DODESHIRUAMMONIUMU montmorillonite clay, and dimethyl dioctadecyl ammonium MOMMORINAITOKURE, etc. is mentioned.

[0037] As an antiperspirant, the antiperspirant chosen from an aluminum chloro hydrate, an

aluminum chloride, an aluminum sesquichloro hydrate, zirconyl hydroxy chloride, aluminum zirconium hydroxy chloride, an aluminum zirconium glycine complex, etc. is mentioned.

[0038] As an ultraviolet ray absorbent, benzoic-acid system ultraviolet ray absorbents, such as p aminobenzoic acid, Salicylic-acid system ultraviolet ray absorbents, such as anthranilic-acid system ultraviolet ray absorbents, such as methyl ortho aminobenzoate, and a methyl salicylate, Benzophenone system ultraviolet ray absorbents, such as cinnamic-acid system ultraviolet ray absorbent [, such as Para methoxycinnamic acid octyl,], 2, and 4-dihydroxy benzophenone, Dibenzoylmethane system ultraviolet ray absorbents, such as urocanic acid system ultraviolet ray absorbent [, such as urocanic acid ethyl] and 4-t-butyl-4'-methoxy-dibenzoylmethane, etc. are mentioned. As an ultraviolet absorption dispersion agent, fine particles which carry out absorption dispersion of the ultraviolet rays, such as particle titanium oxide, particle iron content titanium oxide, a particle zinc oxide, particle cerium oxide, and those complex, are mentioned.

[0039] As a moisturizer, there are a glycerol, a sorbitol, propylene glycol, dipropylene glycol, 1, 3-butylene glycol, a glucose, xylitol, maltitol, a polyethylene glycol, hyaluronic acid, chondroitin sulfate, pyrrolidone carboxylate, polyoxyethylene methyl glucoside, polyoxypropylene methyl glucoside, etc.

[0040] As antimicrobial antiseptics, parahydroxybenzoic acid alkyl ester, a benzoic acid, a sodium benzoate, a sorbic acid, sorbic acid potassium salt, phenoxyethanol, etc. have a benzoic acid, a salicylic acid, a carbolic acid, a sorbic acid, parahydroxybenzoic acid alkyl ester, the PARAKURORU metacresol, hexachlorophene, a benzalkonium chloride, chlorination chlorhexidine, trichlorocarbanilide, an admiration light corpuscle, phenoxyethanol, etc. as an antimicrobial agent.

[0041] As an anti-oxidant, as a pH regulator, a tocopherol, butylhydroxyanisole, dibutylhydroxytoluene, phytic acid, etc. As a chelating agent, a lactic acid, a citric acid, a glycolic acid, a succinic acid, a tartaric acid, dl-malic acid, potassium carbonate, a sodium hydrogencarbonate, an ammonium hydrogencarbonate, etc. As a refrigerant, an alanine, a disodium edetate salt, sodium polyphosphate, sodium metaphosphate, a phosphoric acid, etc. As anti-inflammatory agents, such as L-menthol and camphor, allantoin, glycyrrhizic acid and its salt, glycyrrhetic acid and glycyrrhetic acid stearyl, tranexamic acid, an azulene, etc. are mentioned.

[0042] As a component for lustrous skin, whitening agents, such as a placenta extract, arbutin, a glutathione, and a creeping saxifrage extract, Cell activators, such as royal jelly, an admiration light corpuscle, a cholesterol derivative, and extract from hemolysed blood of young calves, A surface deterioration improvement agent, a nonylic acid crack nil amide, nicotinic-acid benzyl ester, Nicotinic-acid beta-butoxy ethyl ester, capsaicin, a zingerone, Cantharides tincture, ichthammol, caffeine, a tannic acid, alpha-borneol, Tocopherol nicotinate, inositol hexanicotinate, cyclandelate, Antiseborrheic drugs, such as skin astringents, such as circulation accelerators, such as cinnarizine, tolazoline, acetylcholine, verapamil, cepharanthin, and gamma-orizanol, a zinc oxide, and a tannic acid, sulfur, and a CHIAN trawl, etc. are mentioned.

[0043] As vitamins, vitamin A oil, retinol, retinol acetate, Vitamin A, such as retinol palmitate, a riboflavin, riboflavin tetrabutylate, Vitamins B2, such as flavin adenine dinucleotide, pyridoxine hydrochloride, Vitamins B6, such as pyridoxine dioctanoate and pyridoxine tripalmitate Vitamin B, such as vitamin B12 and its derivative, vitamin B 15, and its derivative L-ascorbic acid, L-ascorbic acid dipalmitate ester, L-ascorbic acid-2-sodium sulfate, vitamin C, such as L-ascorbic acid phosphoric-acid diester JIKARIUMU Vitamin D, such as ergocalciferol and

cholecalciferol The alpha-tocopherol, the beta-tocopherol, the gamma-tocopherol, the acetic-acid dl-alpha-tocopherol, Vitamin E, such as nicotinic-acid dl-alpha-tocopherol and succinic-acid dl-alpha-tocopherol Nicotinic acids, such as vitamin H, vitamin P, a nicotinic acid, nicotinic-acid benzyl, and nicotinamide There are pantothenic acid, such as calcium pantothenate, D-pant thenyl alcohol, pant thenyl ethyl ether, and acetyl pant thenyl ethyl ether, a biotin, etc.

[0044] As amino acid, estradiol, ethenyl estradiol, etc. are mentioned as hormone, such as a deoxyribonucleic acid, as nucleic acids, such as a glycine, a valine, a leucine, an isoleucine, a serine, a threonine, a phenylalanine, an arginine, a lysine, an aspartic acid, glutamic acid, a cystine, a cysteine, a methionine, and a tryptophan.

[0045] As a high molecular compound for hair immobilization, both sexes, anionic, cationicity, Each nonionic high molecular compound is mentioned. A polyvinyl pyrrolidone, vinyl pyrrolidone / vinyl acetate copolymer, Acid vinyl ether system high molecular compounds, such as a polyvinyl-pyrrolidone system high molecular compound, and the methyl vinyl ether / maleic-anhydride alkyl half ester copolymer, Acid polyvinyl acetate system macromolecules, such as vinyl acetate / crotonic-acid copolymer, Acid acrylic high molecular compounds, such as an acrylic acid / alkyl (meta) acrylate copolymer, and an acrylic acid (meta) / alkyl (meta) acrylate / alkyl acrylamide copolymer, (Meta) N-methacryloyl ethyl-N and N-dimethylannmonium and alpha-N-methyl carboxy betaine / alkyl (meta) acrylate copolymer, Both-sexes acrylic high molecular compounds, such as hydroxypropyl (meta) acrylate / butylamino ethyl methacrylate / acrylic-acid octyl amide copolymer, are mentioned. Moreover, natural origin high molecular compounds, such as a cellulose or its derivative, a keratin and a collagen, or its derivative, can also be used suitably.

[0046] With the emulsification cosmetics in this invention, ultraviolet-rays defense cosmetics, such as hair cosmetics, such as makeup cosmetics, such as skin care cosmetics, such as face toilet, a milky lotion, a cream, cleansing cream, a pack, a charge of a massage, a cleaning agent, a deodorant, a hand cream, and a lip cream, a makeup substrate, face powder, facial liquid foundation, rouge, eye shadow, mascara, an eyeliner, an eye blow, and a lip stick, a shampoo, a rinse, a treatment, and a set agent, antiperspirant cosmetics, a sunscreen milky lotion, and sun-block cream etc. mentioned

[0047] Moreover, as a product gestalt of these cosmetics, various gestalten, such as the shape of the shape of the shape of the shape of the shape of the shape of the shape of the shape of a liquid, a milk liquid, and a cream and a solid and a paste, gel, powder, and a press and a multilayer and a mousse and a spray and a stick, can be chosen.

[0048]

[Example] This invention is not limited by this although this invention is further explained in full detail according to an example below. In addition, unless it refuses especially, "% of the weight" is meant "%" indicated below. Moreover, the following allyl compound ether derivative was used as a raw material of a triglycerol derivative. Furthermore, M and a Me₂SiO radical were carried out for 1/2 Me₃SiO, MH notation of DH and the 1/2 Me₂HSiO was carried out for D and a MeHSiO radical, and it wrote MR and DR [the unit which denaturalized the methyl group in M and D (Me) by Substituent R], respectively.



[0049] (Example 1 of manufacture) Methyl-hydrogen-polysiloxane 200g [of an average empirical formula (M2D10DH5)], 360g [of allyl compound ether derivatives], and isopropyl alcohol (IPA) 225g and 0.3g of IPA solutions of 0.5% of chloroplatinic acid were taught to the reactor, and it was made to react to the bottom of reflux of a solvent for 8 hours. Reaction mixture was moved to the autoclave after cooling, subsequently Raney nickel catalyst 40g was added, hydrogen was introduced, and it was made to react at 120 degrees C for 3 hours. Hydrogen pressure was held to 0.5MPa(s) during the reaction. Taught the reaction mixture which filtered the catalyst again to the reactor, added 72g of 0.1 N/HCl water solutions, hydrolysis was made to perform at 70 degrees C for 3 hours, and the acetone was made to distill. After adding 15g of 5%-sodium bicarbonate water and making it neutralize, reduced pressure distilling off (– 110 degrees C / 400Pa) was performed, and the organopolysiloxane (siloxane compound 1) expressed with average empirical formula M2D10DR*15 was obtained by filtering. R*1 [however,] –C3H6OCH [CH2OCH2CH(OH) CH2OH]2 – this product – light yellow – transparent – it was liquefied, and viscosity was 180 Pa–S (25 degrees C), and the refractive index was 1.464 (25 degrees C).

[0050] (Example 2 of manufacture) Methyl-hydrogen-polysiloxane 350g [of an average empirical formula (M2D30DH20)], 700g [of allyl compound ether derivatives], and 1-hexa decene 90g, isopropyl alcohol (IPA) 340g, and 0.6g of IPA solutions of 0.5% of chloroplatinic acid were taught to the reactor, and it was made to react to the bottom of reflux of a solvent for 8 hours. Reaction mixture was moved to the autoclave after cooling, subsequently Raney nickel catalyst 75g was added, hydrogen was introduced, and it was made to react at 120 degrees C for 3 hours. Hydrogen pressure was held to 0.5MPa(s) during the reaction. Taught the reaction mixture which filtered the catalyst again to the reactor, and added 140g of 0.1N/HCl water solutions, hydrolysis was made to perform at 70 degrees C for 3 hours, and the acetone was made to distill. After adding 26g of 5%-sodium bicarbonate water and neutralizing, reduced pressure distilling off (– 110 degrees C / 400Pa) was performed, and the organopolysiloxane (siloxane compound 2) expressed with average empirical formula M2D30DR*116D1–hexa decene 4 was obtained by filtering. this product – light yellow – transparent – it was liquefied, and viscosity was 150 Pa–S (25 degrees C), and the refractive index was 1.459 (25 degrees C).

[0051] (Example 3 of manufacture) Methyl-hydrogen-polysiloxane 480g [of an average empirical formula (M2D60DH3)], 130g [of allyl compound ether derivatives], and isopropyl alcohol (IPA) 180g and 0.3g of IPA solutions of 0.5% of chloroplatinic acid were taught to the reactor, and it was made to react to the bottom of reflux of a solvent for 8 hours. Reaction mixture was moved to the autoclave after cooling, subsequently Raney nickel catalyst 40g was added, hydrogen was introduced, and it was made to react at 120 degrees C for 3 hours. Hydrogen pressure was held to 0.5MPa(s) during the reaction. Taught the reaction mixture which filtered the catalyst again to the reactor, and added 26g of 0.1 N/HCl water solutions, it was made to hydrolyze at 70 degrees C for 3 hours, and the acetone was made to distill. After adding 6g of 5%-sodium bicarbonate water and neutralizing, reduced pressure distilling off (– 110 degrees C / 400Pa) was performed, and the organopolysiloxane (siloxane compound 3) expressed with average empirical formula M2D60DR*13 was obtained by filtering. this product – light yellow –

transparent -- it was liquefied, and viscosity was 12.0 Pa-S (25 degrees C), and the refractive index was 1.417 (25 degrees C).

[0052] (Example 4 of manufacture) Methil-hydrogen-polysiloxane 400g [of an average empirical formula (MH2D60)], 100g [of allyl compound ether derivatives], and isopropyl alcohol (IPA) 150g and 0.2g of IPA solutions of 0.5% of chloroplatinic acid were taught to the reactor, and it was made to react to the bottom of reflux of a solvent for 8 hours. Reaction mixture was moved to the autoclave after cooling, subsequently Raney nickel catalyst 32g was added, hydrogen was introduced, and it was made to react at 120 degrees C for 3 hours. Hydrogen pressure was held to 0.5MPa(s) during the reaction. Taught the reaction mixture which filtered the catalyst again to the reactor, and added 20g of 0.1 N/HCl water solutions, it was made to hydrolyze at 70 degrees C for 3 hours, and the acetone was made to distill. After adding 4g of 5%-sodium bicarbonate water and neutralizing, reduced pressure distilling off (- 110 degrees C / 400Pa) was performed, and the organopolysiloxane (siloxane compound 4) expressed with average empirical formula MR*12D60 was obtained by filtering. this product -- light yellow -- transparent -- it was liquefied, and viscosity was 10.0 Pa-S (25 degrees C), and the refractive index was 1.413 (25 degrees C).

[0053] (Example 5 of manufacture) a reactor -- methil-hydrogen-polysiloxane 300g of an average empirical formula (M2D27DH3), 130g of allyl compound ether derivatives, and the oleyl polyoxypropylene-3-allyl compound ether -- [-- the product made from Japanese Emulsifier -- RG-1252]60g, isopropyl alcohol (IPA) 150g, and 0.2g of IPA solutions of 0.5% of chloroplatinic acid were prepared, and it was made to react to the bottom of reflux of a solvent for 8 hours. Reaction mixture was moved to the autoclave after cooling, subsequently Raney nickel catalyst 32g was added, hydrogen was introduced, and it was made to react at 120 degrees C for 3 hours. Hydrogen pressure was held to 0.5MPa(s) during the reaction. Taught the reaction mixture which filtered the catalyst again to the reactor, and added 26g of 0.1 N/HCl water solutions, it was made to hydrolyze at 70 degrees C for 3 hours, and the acetone was made to distill. After adding 6g of 5%-sodium bicarbonate water and neutralizing, reduced pressure distilling off (- 110 degrees C / 400Pa) was performed, and the organopolysiloxane (siloxane compound 5) expressed with average empirical formula M2D27DR*12DR*2 was obtained by filtering. However, R*2 is -C3H6O(C3H6O)3C18H35. this product -- light yellow -- transparent -- it was liquefied, and viscosity was 9.0 Pa-S (25 degrees C), and the refractive index was 1.422 (25 degrees C).

[0054] (Example 6 of manufacture) Methil-hydrogen-polysiloxane 360g [of an average empirical formula (M2D40DH8)], 130g [of allyl compound ether derivatives], and 1-hexa decene 120g, isopropyl alcohol (IPA) 180g, and 0.3g of IPA solutions of 0.5% of chloroplatinic acid were taught to the reactor, and it was made to react to the bottom of reflux of a solvent for 8 hours. Reaction mixture was moved to the autoclave after cooling, subsequently Raney nickel catalyst 40g was added, hydrogen was introduced, and it was made to react at 120 degrees C for 3 hours. Hydrogen pressure was held to 0.5MPa(s) during the reaction. Taught the reaction mixture which filtered the catalyst again to the reactor, and added 26g of 0.1 N/HCl water solutions, it was made to hydrolyze at 70 degrees C for 3 hours, and the acetone was made to distill. After adding 6g of 5%-sodium bicarbonate water and neutralizing, reduced pressure distilling off (- 110 degrees C / 400Pa) was performed, and the organopolysiloxane (siloxane compound 6) expressed with average empirical formula M2D40DR*13D1-hexa decene 5 was obtained by filtering. this product -- light yellow -- transparent -- it was liquefied, and viscosity was 11.0 Pa-S (25 degrees C), and the refractive index was 1.413 (25 degrees C).

[0055] Examples 1-6 and the examples 1-4 of a comparison: The cleaning agent constituent

was prepared by mixing the presentation shown in the cleaning agent constituent table 1.

[0056]

[Table 1]

成分	実施例1	実施例2	実施例3	実施例4	実施例5	実施例6	比較例1	比較例2	比較例3	比較例4
POE (10) ソルビタンモノラウレート (注1)	30	←	←	←	←	←	30	←	←	←
塩化ナトリウム	1	←	←	←	←	←	←	←	←	←
精製水	49	←	←	←	←	←	69	49	←	←
シロキサン化合物1	20	←	20	←	←	←	←	←	←	←
シロキサン化合物2	←	20	←	←	←	←	←	←	←	←
POE (8) オレイルエーテル (注2)	←	←	30	←	←	←	←	←	←	←
POE (50) 硬化ヒマシ油 (注3)	←	←	←	30	←	←	←	←	←	←
POE (10) モノオレート (注4)	←	←	←	←	30	←	←	←	←	←
グリセロールモノラウレート (注5)	←	←	←	←	←	30	←	←	←	←
ポリエーテル変性シリコーン1 (注6)	←	←	←	←	←	←	←	20	←	←
ポリエーテル変性シリコーン2 (注7)	←	←	←	←	←	←	←	←	20	←
ポリエーテル変性シリコーン3 (注8)	←	←	←	←	←	←	←	←	←	20

(注1) ポリオキシエチレン (10) ソルビタンモノラウレート: (三洋化成工業 (株) 社製)

(注2) ポリオキシエチレン (8) オレイルエーテル: EMALLES 510 (日本エマルジョン (株) 社製)

(注3) ポリオキシエチレン (50) 硬化ヒマシ油: HCO-50 (日光ケミカルズ (株) 社製)

(注4) ポリオキシエチレン (10) モノオレート: EMALLEX OE-10 (日本エマルジョン (株) 社製)

(注5) グリセロールモノラウレート: (三洋化成工業 (株) 社製)

(注6) ポリエーテル変性シリコーン1: KF6011 (信越化学工業 (株) 社製) 分子量4500

(注7) ポリエーテル変性シリコーン2: KF6013 (信越化学工業 (株) 社製) 分子量4000

(注8) ポリエーテル変性シリコーン3: KF6017 (信越化学工業 (株) 社製) 分子量6000

[0057] The use test in the case of dropping the appearance and lip stick was performed, the following criteria of the mileage breadth at the time of spreading, the earliness of concordance with dirt, dirt omission, and the back skin estimated admiration gently, and each cleaning agent constituent was judged in the average mark. The result is as having been shown in Table 2.

[0058] (The evaluation approach)

(Appearance)

O - transparency ** - Translucent x-opacity (a feeling of use, and usability)

[Valuation basis]

five point: -- very much -- four fitness: three fitness: -- usually -- two point: -- a little -- defect 1 point: -- a defect [a judgment]

O Less than [or more / the 4.5 or more :average mark O:average-mark / or more 3.5 / less than 4.5 / **:average mark / 2.5 3.5] x : the less than 2.5 average mark [0059]

[Table 2]

	実施例1	実施例2	実施例3	実施例4	実施例5	実施例6	比較例1	比較例2	比較例3	比較例4
外観	◎	○	◎	◎	◎	◎	△	△	△	△
塗布時ののび広がり	◎	○	◎	◎	◎	◎	x	○	○	△
汚れとのなじみの早さ	◎	◎	◎	◎	◎	◎	x	△	△	△
汚れ落ち	◎	◎	◎	◎	◎	◎	△	△	△	△
後肌のしっとり感	◎	◎	◎	◎	◎	◎	x	△	△	△

[0060] Compared with the examples 1-4 of a comparison, the concordance of early and dirt omission was also very good to the dirt of the Kamiguchi red with a transparent appearance, the mileage breadth at the time of spreading was also still better, the cleaning agent constituent of the examples 1-6 of this invention also carried out the back skin gently, and its feeling of use was very good so that clearly from the result of Table 2.

[0061]

Example 7: Makeup remover (component) (%)

1. POE (10) Sorbitan Monolaurate 10.02. Siloxane compound 1 20.03. Sorbitol 10.04.

Carrageenan 0.55. Glycerol 5.06. Sodium citrate 0.57. antiseptics ** Amount 8. perfume **

Amount 9. purified water 54.0 (the manufacture approach)

A: It reached component 1-7, 9 was added, and it dissolved in homogeneity.

B: The component 8 was added to A and the makeup remover was obtained.

When foundation was removed using the makeup remover obtained as mentioned above, concordance with foundation was also good, dirt omission was also very good, the mileage at the time of use was also light, and the back skin was also carried out gently and was a makeup remover also with a very sufficient feeling of usability and use.

[0062]

Example 8: Hair makeup remover (component) (%)

1. Polyoxyethylene (15) Isocetyl Ether (Notes 1) 10.02. Siloxane compound 2 20. 03.1, 3-butylene glycol 10.04. Glycerol 10.05. Carrageenan 0.56. Sodium chloride 0.57. ***** Amount 8. perfume ** Amount 9. purified water 49.0 (notes 1) polyoxyethylene (15) isocetyl ether : (Sanyo Chemical Industries [, Ltd.], Ltd. make)

(The manufacture approach)

A: It reached component 1-7, 9 was added, and it dissolved in homogeneity.

B: The component 8 was added to A and the hair makeup remover was obtained.

When hair was washed using the hair makeup remover obtained as mentioned above, the top where a hair makeup is good, dirt omission also had very good concordance with sebum dirt well, and the mileage at the time of use also had it, it did not have the stickiness after use, either, was carrying out gently, and a feeling of usability and use was also a very good hair makeup remover. [light]

[0063]

Example 9: Charge of washing its face (component) (%)

1. Polyoxyethylene (6) Lauryl Ether (Notes 1) 5.02. Siloxane compound 2 10.03. Ethanol 10.04. Lauryl dimethylamine oxide (notes 2) 2.05. Propylene glycol 3.06. Sodium citrate 0.57. Antiseptics ** Amount 8. perfume ** Amount 9. purified water 69.5 (notes 1) polyoxyethylene (6) lauryl ether: Peg Norian L-6 (Toho Chemical Industry [Co., Ltd.] Co., Ltd. make) (notes 2) Lauryl dimethylamine oxide : uni-safe A-LM (Nippon Oil & Fats [Co., Ltd.] Co., Ltd. make)

(The manufacture approach)

A: It reached component 1-7, 9 was added, and it dissolved in homogeneity.

B: The component 8 was added to A and the charge of washing its face was obtained.

When the charge of washing its face obtained as mentioned above was used, concordance with cosmetics or sebum dirt was good, dirt omission also had it, the mileage at the time of use also had it, the stickiness after use did not have it, either, the back skin was also carried out gently, there was also no stickiness, and a feeling of usability and use was also a very good charge of washing its face. [light] [very good]

[0064]

Example 10: Drop [makeup] (component). (%)

1. Polyoxyethylene (6) Sorbitan Monolaurate (Notes 1) 5.02. Siloxane compound 1 5.03. Siloxane compound 2 15.04. Ethanol 10.05. Glycerol 2.06. Dipropylene glycol 3.07. Sodium glutamate 0.58. antiseptics ** Amount 9. perfume ** Amount 10. purified water 59.5 (notes 1) polyoxyethylene (6) sorbitan monolaurate : (Sanyo Chemical Industries [, Ltd.], Ltd. make)

(The manufacture approach)

A: It reached component 1-8, 10 was added, and it dissolved in homogeneity.

B: The component 9 was added to A and it obtained makeup dropping.

When it used makeup dropping [which was obtained as mentioned above], concordance with cosmetics or sebum dirt is good, dirt omission also has it, the mileage at the time of use also

has it, the stickiness after use does not have it, either, the back skin was also carried out gently, there was no stickiness, and a feeling of usability and use was also very good makeup dropping.
[light] [very good]

[0065]

Example 11: Polyhydric-alcohol emulsification cosmetics in an oil (component) (%)

1. Bridge Formation Mold Dimethylpolysiloxane (Notes 1) 30.02. Decamethyl cyclopentasiloxane 15.03. Dimethylpolysiloxane (6mm²/second (25 degrees C)) 7.04. Siloxane compound 3 3.05. Dimethyl distearyl ammonium hectorite 2.06. Antiseptics ** Amount 7. perfume ** Amount 8. sodium chloride 0.059.1, 3-butylene glycol 42.95 (notes 1) bridge-formation mold dimethylpolysiloxane: KSG15 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(The manufacture approach)

A: It reached component 1-5 and 7 was mixed to homogeneity.

B: Components 6, 8, and 9 were mixed.

C: B was added to A and it emulsified to homogeneity.

It was checked that they are the polyhydric-alcohol emulsification cosmetics in a nonaqueous oil with sufficient stability which the polyhydric-alcohol emulsification cosmetics in an oil obtained as mentioned above have light mileage breadth, and have neither stickiness nor oiliness, and the back skin carried out gently.

[0066]

Example 12: Polyhydric-alcohol emulsification HOHO red in a solid-like oil (component) (%)

1. Bridge Formation Mold Dimethylpolysiloxane (Notes 1) 5.02. Decamethyl cyclopentasiloxane 5.03. Dimethylpolysiloxane (6mm²/second (25 degrees C)) 19.74. Cetyl iso OKUTANETO 15.05. Paraffin wax (melting point of 80 degrees C) 12.06. Siloxane compound 3 3.07. Dimethyl distearyl ammonium hectorite 0.28. Hydrophobing processing fine particles 25.09. Sodium citrate 0.110. Antiseptics ** Amount 11. perfume ** An amount 12.1, 3-butylene glycol 15.0 (notes 1) bridge-formation mold dimethylpolysiloxane: KSG15 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(The manufacture approach)

A: It reached component 1-7, 11 was heated at 80 degrees C, and it mixed to homogeneity.

B: The component 8 was added to A and it distributed to homogeneity.

C: In addition to the components 9, 10, and B beforehand heated at 80 degrees C, 12 was emulsified, and it slushed into the metal dish and cooled.

It was checked that it is polyhydric-alcohol emulsification HOHO in a solid-like oil with sufficient stability which the polyhydric-alcohol emulsification HOHO red in a solid-like oil obtained as mentioned above has light mileage breadth, and has neither stickiness nor oiliness, and the back skin carried out gently.

[0067]

Example 13: Cream-like lip stick (component) (%)

1. Palmitic Acid / Ethylhexanoic Acid Dextrin (Notes 1) 9.02. TORIISO octanoic-acid glyceryl 22.03. Bentonite 0.74. Siloxane compound 4 1.55. Decamethyl cyclopentasiloxane 42.06.1, 3-butylene glycol 5.07. sodium chloride 0.58. purified water 19.39. color pigment ** An amount (notes 1) palmitic acid / ethylhexanoic-acid dextrin: LEO pearl TT (Chiba Flour Milling [Co., Ltd.] Co., Ltd. make)

(The manufacture approach)

A: A part of component 1, component 2, and components 3-5 were mixed, and it dissolved.

B: The component 9 was mixed in the remainder of a component 2, and the roller distributed in

it.

C: B was added to A and it mixed to homogeneity.

D: Components 6-8 were mixed and warmed.

E: D was added to C and emulsified.

The lip stick obtained as mentioned above is a cream-like lip stick of the W/O mold excellent in makeup durability, and it was checked that mileage breadth is light and there is neither stickiness nor oiliness.

[0068]

Example 14: Eyeliner (component) (%)

1. Octamethylcyclotetrasiloxane 53.52. Siloxane compound 4 3.03. Silicone reticulated resin (notes 1) 15.04. Dimethyl distearyl ammonium hectorite 3.05. Siliconization black oxide of iron (notes 2) 10.06.1, a 3-butylene-glycol 5.07. sodium sulfate 0.58. Antiseptics ** Amount 9. purified water As opposed to black oxide of iron 10.0 (notes 1) silicone reticulated resin: -- $[\text{Me}_3\text{SiO } 1/2] / [\text{SiO}_2]$ ratio -- 50%-D5 solution (notes 2) siliconization black-oxide-of-iron [of the silicone reticulated compound of 0.8]: -- What was heat-treated after 2% of methyl-hydrogen-polysiloxane addition (the manufacture approach)

A: Components 1-4 were mixed, the component 5 was added, and mixed distribution was carried out at homogeneity.

B: Components 6-9 were mixed.

C: B was ****(ed) and emulsified to A and the eyeliner was obtained.

The eyeliner obtained as mentioned above has light mileage, and it is easy to draw it, it is cool, is felt refreshed, when it is the feeling of use without stickiness, it does not have change by temperature or the passage of time, usability and stability are very excellent, both a water resisting property and perspiration resistance were excellent, and it was checked that makeup **** is also very good.

[0069]

Example 15: Eye shadow (component) (%)

1. Decamethyl Cyclopentasiloxane 15.02. Dimethylpolysiloxane (6mm²/second (25 degrees C)) 10.03. Siloxane compound 4 2.04. PEG(10) RAURIRUE-Tell 0.55. Siliconization chrome oxide (notes 1) 6.26. Siliconization ultramarine blue (notes 1) 4.07. siliconization titanium covering mica (notes 1) 6.08. sodium chloride 2.09. Propylene glycol 8.010. Antiseptics ** Amount 11. perfume ** Amount 12. purified water 46.3 (notes 1) siliconization; what was heat-treated after 3% of methyl-hydrogen-polysiloxane addition to fine particles (the manufacture approach)

A: Components 1-4 were mixed, components 5-7 were added, and it distributed to homogeneity.

B: It reached component 8-10 and the homogeneity dissolution of 12 was carried out.

C: The component 11 was added and eye shadow was obtained, after ****(ing) and emulsifying B to A, the bottom of stirring, and.

while mileage breadth is light, and the eye shadow obtained as mentioned above has neither oiliness nor powderiness, is fresh and giving the clean feeling of use -- gently -- carrying out -- **** -- a water resisting property, water repellence, and perspiration resistance -- good -- also having -- it being good, makeup being hard to come off, and there being no change by temperature or the passage of time, and excelling also in stability was checked.

[0070]

Example 16: Suntan milky lotion (component) (%)

1. Emulsifier Constituent (Notes 1) 6.02. Dimethylpolysiloxane (6mm²/second (25 degrees C)) 49.03.1, 3-butylene glycol 5.04. Sodium dehydroacetate 0.25. Antioxidant ** Amount 6.

antiseptics ** Amount 7. perfume ** Amount 8. purified water 39.8 (notes 1) Emulsifier constituent a. siloxane compound 4 10.0 weight sections b. dioctadecyl dimethylammonium salt denaturation montmorillonite 10.0 weight section c. ethanol The 40.0 weight sections (the manufacture approach)

A: Component a was dissolved in c and Component b was added.

B: After stirring A in DISUPA for 1 hour, ethanol was removed by the evaporator.

C: B was dried at 50 degrees C one whole day and night, and the emulsifier constituent of a component 1 was obtained.

D: The component 1 and component 2 which were obtained by C were mixed.

E: It reached component 3-6 and 8 was mixed to homogeneity.

F: E was ****(ed) and emulsified to D under stirring, the component 7 was added, and the suntan milky lotion was obtained.

Mileage breadth being light, a water resisting property and makeup **** being also good [the suntan milky lotion obtained as mentioned above has a fine texture, and], while giving the clean feeling of use which there is neither stickiness nor oiliness, and does not try not to carry out gently but is spread, and there being no change by temperature or the passage of time, and excelling also in stability was checked.

[0071]

Example 17: SANKATTO cream (component) (%)

1. Decamethyl Cyclopentasiloxane 17.52. Acrylic silicone resin / decamethyl cyclopentasiloxane (notes 1) 12.03. TORISO octanoic-acid glyceryl 5.04. Para methoxycinnamic acid octyl 6.05. Bridge formation mold polyether denaturation silicone (notes 2) 5.06. Siloxane compound 6 1.07. Lipophilic-ized processing zinc oxide 20.08. Sodium chloride 0. 59.1, 3-butylene glycol 2.010. Antiseptics ** Amount 11. perfume ** Amount 12. purified water 31.0 (notes 1) acrylic silicone resin / decamethyl cyclopentasiloxane: KP545 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make) (notes 2) Bridge formation mold polyether denaturation silicone : KSG21 (Shin-Etsu Chemical Co., Ltd.)

Shrine make

(The manufacture approach)

A: The component 2 was added to a part of component 1, it was made homogeneity, the component 7 was added, and the bead mill distributed.

B: Components 3-6 were mixed with the remainder of a component 1 to homogeneity.

C: It reached component 8-10, 12 was mixed, and it dissolved in homogeneity.

D: C was added and emulsified to B, A and a component 11 were added, and the SANKATTO cream was obtained.

the SANKATTO cream obtained as mentioned above -- stickiness -- there is nothing -- mileage breadth -- light -- moreover -- a feeling of adhesion -- excelling -- also being subsided -- it was good, and it is finished, and it comes out, makeup **** is also very excellent, and the glossy very stable thing was checked also to temperature or the passage of time.

[0072]

Example 18: Suntan cream (component) (%)

1. Decamethyl Cyclopentasiloxane 15.02. Dimethylpolysiloxane (6mm²/second (25 degrees C)) 5.03. Stearyl denaturation acrylic silicone (notes 1) 0.54. Siloxane compound 5 6.05. Palmitic acid 0.26. Dimethyl octyl p aminobenzoic acid A 0.57.4-t-butyl-4'-methoxy-dibenzoylmethane 0.58. kaolin 0.59. Red ocher 0.210. Yellow oxide of iron 0.311. Black oxide of iron 0.112. titanium oxide coated mica 1.013. Sodium L-glutamate monohydrate 3. 014.1, 3-butylene glycol 5.015.

dioctadecyl dimethyl ammonium chloride 0.116. antioxidant ** Amount 17. antiseptics ** Amount 18. perfume ** Amount 19. purified water 62.1 (the manufacture approach)
(notes 1) Stearyl denaturation acrylic silicone : KP561 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

A: The heating dissolution of 17 was carried out at components 1-7 and 16 lists.

B: After heating stirring, components 8-12 were added and distributed processing of a part of components 15 and 19 was carried out.

C: The remainder of components 13, 14, and 19 was dissolved in homogeneity, and it mixed with B.

D: Under stirring, C was ****(ed) and emulsified to A, it cooled to it, the component 18 was added further, and the suntan cream was obtained.

While giving the clean feeling of use which the suntan cream obtained as mentioned above has a fine texture, mileage breadth is light, and there is neither stickiness nor oiliness, and does not try not to carry out gently but is spread, it excelled in a feeling of a fit, and makeup **** was also good, and there is no change of separation, condensation of fine particles, etc., and excelling also in stability was checked by temperature and the passage of time.

[0073]

Example 19: The Sun Katt milky lotion (component) (%)

1. Decamethyl Cyclopentasiloxane 3.02. Dimethylpolysiloxane (6mm²/second (25 degrees C)) 5.03. TORIISO octanoic-acid glyceryl 5.04. Siloxane compound 3 1.05. Bridge formation mold polyether denaturation silicone (notes 1) 3.06. Titanium oxide / decamethyl cyclopentasiloxane dispersion liquid (notes 2) 25.07. zinc oxide / decamethyl cyclopentasiloxane dispersion liquid (notes 3) 35.08. dipropylene glycol 3.09. sodium citrate 0.510. Antiseptics ** Amount 11. perfume ** Amount 12. purified water 19.5 (notes 1) bridge-formation mold polyether denaturation silicone: KSG-21 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make) (notes 2) Titanium oxide / decamethyl cyclopentasiloxane dispersion liquid : SPD-T1S (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make) (notes 3) A zinc oxide / decamethyl cyclopentasiloxane dispersion liquid : SPD-Z1S (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make) (The manufacture approach)

A: Components 1-5 were mixed and it mixed to homogeneity.

B: It reached component 8-10, 12 was mixed, and it dissolved.

C: B was added and emulsified to A, components 6, 7, and 11 were added, and the Sun Katt milky lotion was obtained.

the Sun Katt milky lotion obtained as mentioned above -- stickiness -- there is nothing -- mileage breadth -- light -- moreover -- a feeling of adhesion -- excelling -- also being subsided -- it was good, and it is finished, and it comes out, makeup **** is also very excellent, and the glossy very stable thing was checked also to temperature or the passage of time.

[0074]

Example 20: Foundation (component) (%)

1. Decamethyl Cyclopentasiloxane 45.02. Dimethylpolysiloxane (6mm²/second (25 degrees C)) 5.03. Siloxane compound 4 1.54. Siloxane compound 6 0.55. Dimethyl distearyl ammonium hectorite 4.06. Hydrophobing processing titanium oxide (notes 1) 10.07. Hydrophobing processing talc (notes 1) 6.08. Hydrophobing processing mica (notes 1) 6.09. Hydrophobing processing red ocher (notes 1) 1.610. Hydrophobing processing yellow oxide of iron (notes 1) 0.711. Hydrophobing processing black oxide of iron (notes 1) 0.212. Dipropylene glycol 5.013.

Parahydroxybenzoic acid methyl ester A 0.314.2-amino-2-methyl-1,3-propanediol 0.215. hydrochloric acid 0.116. perfume ** Amount 17. water 13.9 (notes 1) hydrophobing processing: what was heat-treated after adding 2% of methyl hydrogen polysiloxane to fine particles (the manufacture approach)

A: Heating mixing of the components 1-5 was carried out, and components 6-11 were added and it was made homogeneity.

B: It reached component 12-15 and the heating dissolution of 17 was carried out (pH of a drainage system is 9.0).

C: The bottom of stirring, and after ****(ing) and emulsifying B to A and cooling to it, the component 16 was added further, and foundation was obtained.

Mileage breadth being light, the foundation obtained as mentioned above having a fine texture, makeup **** being also good while giving the clean feeling of use which there is neither stickiness nor oiliness, and does not try not to carry out gently but is spread, and there being no change by temperature or the passage of time, and excelling also in stability was checked.

[0075]

Example 21: Liquefied foundation (component) (%)

1. Decamethyl Cyclopentasiloxane 16.02. Dimethylpolysiloxane (6mm²/second (25 degrees C)) 8.03. PARAMETOKISHI cinnamic acid octyl 3.04.12-hydroxy stearin acid 1.05. Siloxane compound 5 2.06. Fluorine denaturation silicone (notes 1) 5.07. Spherical silicone resin fine particles (notes 2) 3.08. Fluorine compound processing particle titanium oxide (notes 3) 8.09. Fluorine compound processing mica titanium (notes 3) 1.010. Fluorine compound processing titanium oxide (notes 3) 5.011. Fluorine compound processing red ocher (notes 3) 0.912. Fluorine compound processing yellow oxide of iron (notes 3) 2.013. fluorine compound processing black oxide of iron (notes 3) 1.014. Ethanol 15.015. Glycerol 3.016. Magnesium sulfate 1.017. antiseptics ** Amount 18. perfume ** Amount 19. purified water 25.1 (notes 1) fluorine denaturation silicone: floor line-5 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make) (notes 2) Spherical silicone resin fine particles : KMP590 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(notes 3) Fluorine compound processing : what was covered with the perfluoroalkyl ethyl phosphoric-acid diethanolamine salt 5% (the manufacture approach)

A: Components 7-13 were mixed to homogeneity.

B: Heating mixing of the components 1-6 was carried out at 70 degrees C, A was added and distributed mixing was carried out at homogeneity.

C: After having reached component 14-17, having ****(ed) 19 at 40 degrees C at warming and B, emulsifying and cooling, the component 18 was added further, and liquefied foundation was obtained.

The liquefied foundation obtained as mentioned above does not have stickiness, and mileage breadth is also light, moreover it has the clean high coolness, there is no change by temperature or the passage of time, and it was checked that stability is very excellent.

[0076]

Example 22: Hair cream (component) (%)

1. Decamethyl Cyclopentasiloxane 10.02. Methylphenyl polysiloxane 5.03. Squalane 4.04. Silicone reticulated resin (notes 1) 1.05. JIOREIN acid glyceryl 2.06. Siloxane compound 4 4.07. sorbitol sodium sulfate 2.08. Sodium chondroitin sulfate 1.09. hyaluronate sodium 0.510. Propylene glycol 3.011. Antiseptics 1.512. Vitamin-E acetate 0.113. Antioxidant ** Amount 14. perfume ** Amount 15. purified water 65.9 (notes 1) silicone reticulated resin: [Me₃SiO 1/2] /

[SiO₂] ratio is 50%-D5 solution (the manufacture approach) of the silicone reticulated compound of 0.8.

A: Heating mixing of components 1-6, and 11-13 was carried out.

B: It reached component 7-10 and the heating dissolution of 15 was carried out.

C: A part for the epigenesis 14 which ****(ed) and emulsified B and was cooled was added to A under stirring, and the hair cream was obtained.

while giving the clean feeling of use which the hair cream obtained as mentioned above has light mileage breadth, and there is neither stickiness nor oiliness, and does not try not to carry out gently but is spread -- a water resisting property, water repellence, and perspiration resistance -- it is -- also having -- it being good, and there being no change by temperature or the passage of time, and excelling also in stability was checked.

[0077]

Example 23: Hair cream (component) (%)

1. Silicone Gum Dissolution Article (40,000MPa and Second) 18.02. Silicone reticulated resin (notes 1) 6.03. Tree 2-ethylhexanoic acid glyceryl 8.04. Vaseline 5.05. Stearyl alcohol 2.06. Mono-oleic acid sorbitan 2.07. Siloxane compound 62.08. glycerol 5.09. Sodium chloride 0.510. Perfume ** Amount 11. purified water 51.5 (notes 1) silicone reticulated resin: [Me₃SiO 1/2] / [SiO₂] ratio is 50%-D5 solution (the manufacture approach) of the silicone reticulated compound of 0.8.

A: Heating mixing of the components 1-7 was carried out.

B: component 8- mixed stirring of 9 and 11 was carried out.

C: A was ****(ed) and emulsified to B under stirring, the component 10 was added, and the hair cream was obtained.

While giving the clean feeling of use which the hair cream obtained as mentioned above has light mileage breadth, and has neither stickiness nor oiliness, and does not try not to carry out gently but is spread, gloss and smoothness were given to hair and having the outstanding set effectiveness over hair was checked.

[0078]

Example 24: Moisturization cream (component) (%)

1. Decamethyl Cyclopentasiloxane 10.02. Methylphenyl polysiloxane 3.03. Liquid paraffin 5.04. Steer ROKISHI denaturation silicone (notes 1) 8.05. Siloxane compound 8 2.06. Organopolysiloxane elastomer spherical fine particles (notes 2) 2.57. Hydrophobing silica (notes 3) 2.08. Zinc stearate 2.09. Vitamin-E acetate 3.010. Polyethylene glycol 400 1.011. Sodium lactate 1. 012.1, 3-butylene glycol 5.013. antiseptics ** Amount 14. perfume ** Amount 15. purified water 55.5 (notes 1) steer ROKISHI denaturation silicone: KF-7002 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(notes 2) an organopolysiloxane elastomer -- spherical -- fine-particles;KMP-590 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(notes 3) Hydrophobing silica; Aerosil R972 (product made from Japanese Aerosil)

(The manufacture approach)

A: 9 was mixed to homogeneity at components 1-5 and component 8 list, components 6-7 were added, and it distributed to homogeneity.

B: It reached component 10-13, and 15 was added and it dissolved.

C: It cooled, after ****(ing) and emulsifying B to A, and the component 14 was added, and the moisturization cream was obtained.

Excelling also in the usability and stability which the mileage breadth of the moisturization

cream obtained as mentioned above is also light, it feels refreshed fresh and there is no stickiness, carry out gently and change by temperature or the passage of time does not have, either very much was checked.

[0079]

Example 25: Hand cream (component) (%)

1. Decamethyl Cyclopentasiloxane 30.02. Liquid paraffin 10.03. Amino denaturation silicone gum (amine equivalent 70,000 g/mol) 15.04. siloxane compound 6 4.05. Distearyl dimethyl ammonium chloride 0.86. Vitamin-E acetate 0.17. Polyethylene glycol 4000 1.08. Glycerol 10.09. magnesium aluminum silicate 1.210. Antiseptics ** Amount 11. perfume ** Amount 12. purified water 27.9 (the manufacture approach)

A: components 1 and 3 -- the heating mixing dissolution -- carrying out -- a component 2 and 4-- heating addition of 6 and 10 was carried out.

B: It reached component 7-9 and heating mixing of 12 was carried out.

C: It cooled, after ****(ing) and emulsifying B to A, and the component 11 was added, and the hand cream was obtained.

The hand cream obtained as mentioned above did not have stickiness, mileage breadth is also light, moreover it has the feeling of use carried out gently, the skin was effectively protected from kitchenwork, and it was checked that temperature stability is very excellent.

[0080]

Example 26: O/W hand cream (component) (%)

1. Acrylic Silicone Resin / Decamethyl Cyclopentasiloxane (Notes 1) 10.02. Stearyl denaturation acrylic silicone resin (notes 2) 8.03. Cetanol 1.04. Tori isostearic acid glyceryl 5.05. Stearin acid 3.06. Monostearin acid glyceryl 1.57. Siloxane compound 20.78. sorbitan sesquioleate 0.59. Mono-oleic acid polyoxyethylene sorbitan 1.010. sodium hydroxide (1% water solution) 10.011.1, 3-butylene glycol 5.012. antiseptics ** Amount 13. perfume ** Amount 14. purified water 54.3 (notes 1) acrylic silicone resin / decamethyl cyclopentasiloxane: KP545 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(notes 2) Stearyl denaturation acrylic silicone resin : KP561 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(The manufacture approach)

A: Components 1-9 were mixed and the heating dissolution was carried out.

B: It reached component 10-12, and 14 was mixed and heated.

C: A part for the epigenesis 13 which added B to A, emulsified and was cooled was added, and the O/W hand cream was obtained.

the hand cream obtained as mentioned above -- stickiness -- there is nothing -- mileage breadth -- light -- moreover -- a feeling of adhesion -- excelling -- also being subsided -- it is good and glossy -- it is finished, and it comes out, makeup **** is also very excellent, and it was checked being temperature and that it is very stable also with time.

[0081]

Example 27: Milky lotion (component) (%)

1. Decamethyl Cyclopentasiloxane 15.02. Methylphenyl polysiloxane 5.03. Squalene 5.04. Tetra--2-ethylhexanoic acid pentaerythritol 5.05. Siloxane compound 8 3.06.

Organopolysiloxane elastomer spherical fine particles (notes 1) 2.07. Hydrophobing silica (notes 2) 0.58. Ascorbic-acid magnesium phosphate 1.09. Sodium chloride 1.010. Polyethylene glycol 11000 1.011. Propylene glycol 8.012. Antiseptics ** Amount 13. perfume ** Amount 14. purified water 53.5 (notes 1) organopolysiloxane elastomer spherical fine particles: KMP590 (Shin-Etsu

Chemical [Co., Ltd.] Co., Ltd. make)

(notes 2) Hydrophobing silica : Aerosil R972 (product made from Japanese Aerosil)

(The manufacture approach)

A: Components 1-5 were mixed to homogeneity, components 6 and 7 were added, and it distributed to homogeneity.

B: Components 8-10 were added to the component 14, it dissolved in it, and adding after mixing of the components 11 and 12 was further mixed and carried out to homogeneity.

C: It cooled, after ****(ing) and emulsifying B to A, and the component 13 was added, and the milky lotion was obtained.

The mileage breadth of the milky lotion obtained as mentioned above was also light, and it was checked that the usability which carries out entirely, does not have stickiness and does not have change by temperature or the passage of time is also very excellent also in stability.

[0082]

Example 28: Essence (component) (%)

1. Decamethyl Cyclopentasiloxane 12.02. TORISO octanoic-acid glyceryl 10.03. Siloxane compound 4 2.04. Siloxane compound 5 0.25. Glycerol 10.06. Ascorbic-acid magnesium phosphate salt 3.07. Sodium chloride 2.08. antiseptics ** Amount 9. perfume ** Amount 10. purified water 60.8 (the manufacture approach)

A: Heating mixing of the components 1-4 was carried out.

B: It reached component 5-8, 10 was heated, and the homogeneity dissolution was carried out.

C: A part for the epigenesis 9 which ****(ed) and emulsified B and was cooled was added to A under stirring, and the essence was obtained.

The essence obtained as mentioned above has a fine texture, mileage breadth is light, and there is no stickiness, and it does not try not to carry out gently, but spreads, and temperature and it being changeless with time and excelling also in stability very much were checked.

[0083]

Example 29: Antiperspirant (component) (%)

1. Octamethyl Cyclopentasiloxane 30.02. Siloxane compound 4 1.03. Mono-oleic acid polyoxyethylene sorbitan (20E.O.) The glycine salt of 0.54. aluminum zirconium hydrate tetrachloride 20.05. water 48.5 (the manufacture approach)

A: Components 1-2 were mixed.

B: The component 4 was dissolved in 5 and the component 3 was added.

C: B was ****(ed) and emulsified to A under stirring, and the antiperspirant was obtained.

The antiperspirant obtained as mentioned above did not become not much white, when mileage breadth was light and there was neither stickiness nor oiliness, but while giving the clean feeling of use, there being no change by temperature or the passage of time, and excelling also in stability was checked.

[0084]

Example 30: Cleansing cream (component) (%)

1. Dimethylpolysiloxane (6Mm2/Second (25 Degrees C)) 5.02. Methylphenyl polysiloxane 5.03. Liquid paraffin 8.04. Jojoba oil 2.05. Siloxane compound 4 2.56. Siloxane compound 6 0.57. Dextrin fatty acid ester 0.88. Aluminum monostearate salt 0.29. Aluminum chloride 1.010. Glycerol 10.011. antiseptics ** Amount 12. perfume ** Amount 13. purified water 65.0 (the manufacture approach)

A: Heating mixing of the components 1-8 was carried out.

B: It reached component 9-11 and the heating dissolution of 13 was carried out.

C: A part for the epigenesis 12 which ****(ed) and emulsified B and was cooled was added to A under stirring, and cleansing cream was obtained.

While giving the clean feeling of use which the cleansing cream obtained as mentioned above has a fine texture, mileage breadth is light, and there is neither stickiness nor oiliness, and does not try not to carry out gently but is spread, the cleansing cream effectiveness was also high and temperature and it being changeless with time and excelling also in stability were checked.
[0085]

Example 31: Treatment gel (component) (%)

1. Ethanol 20.02. Silicone compound 3 0.53. TORISO octanoic-acid glyceryl 3.04. Steer ROKISHI denaturation silicone (notes 1) 2.05. Silicone compound powder (notes 2) 8.06. Carboxyvinyl polymer (1% water solution) 20.07. Triethanolamine 0.28. Antiseptics ** Amount 9. perfume ** Amount 10. purified water 46.3 (notes 1) steer ROKISHI denaturation silicone: KF-7002 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)
(notes 2) Silicone compound powder : KSP-100 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(The manufacture approach)

A: Mixed distribution of the components 1-5 was carried out.

B: component 6- 8 and 10 were mixed and it was made homogeneity.

C: A was ****(ed) to B, the component 9 was added, and it mixed to homogeneity.

While giving the clean feeling of use which the treatment gel obtained as mentioned above has light mileage breadth, and there is neither stickiness nor oiliness, and does not try not to carry out gently but is spread, concordance, the temperature which becomes empty, and it being changeless with time and excelling also in stability were checked by the skin.

[0086]

Example 32: Washout type pack cosmetics (component) (%)

1. Dimethylpolysiloxane (6Mm2/Second (25 Degrees C)) 3.02. Siloxane compound 2 2.03. Kaolin 30.04. Carboxyvinyl polymer 0. 45.1, 3-butylene glycol 10.06. Glycerol 20.07. Triethanolamine 0.48. Antiseptics ** Amount 9. perfume ** Amount 10. purified water 34.2 (the manufacture approach)

A: Components 1, 2, and 8 were mixed.

B: After reaching component 4-7 and carrying out homogeneity mixing of 10, mixed stirring of the component 3 was carried out.

C: B was made to add and emulsify A, the component 9 was added further, and washout type [paste-like] pack cosmetics were obtained.

It washed away, during spreading, the pack cosmetics of a type had light mileage breadth, and were excellent in the cleaning effect, after [which was flushed] being obtained as mentioned above, it is the feel which carried out gently, and there is no stickiness and the skin was smooth, and having the feeling of use which was very excellent, and excelling also in stability was checked.

[0087]

Example 33: Deodorant (component) (%)

1. Decamethyl Cyclopentasiloxane 12.02. Dimethylpolysiloxane (6mm2/second (25 degrees C)) 4.03. Siloxane compound 4 1.04. Propylene glycol 31.05. Triclosan 0.16. Glycerol 15.07. sodium chloride 0.18. antiseptics ** Amount 9. perfume ** Amount 10. purified water 36.8 (the manufacture approach)

A: Components 1-3 were mixed.

B: a component 5 -- 4 -- dissolving -- component 6-- 8 and 10 were mixed.

C: B was added and emulsified, stirring A violently and the component 9 was added.

D: The 65 sections and the propellants (n-butane, isobutane, propane mixture) 35 section were added for C to the aerosol can, and the deodorant was obtained.

Having the extraordinary ***** usability which the deodorant obtained as mentioned above does not have stickiness, either, without hanging down even if it uses it for high concentration, is carrying out entirely, and effectiveness maintains was checked.

[0088]

Example 34: O/W/O mold milky lotion (component) (%)

1. Bridge Formation Mold Polyether Denaturation Silicone (Notes 1) 3.02. Siloxane compound 4 1.03. TORIISO octanoic-acid glyceryl 14.04. Bridge formation mold alkyl denaturation silicone compound (notes 2) 5.05. Cane-sugar monostearate 3.06. Glycerol 5. 07.1, 3-butylene glycol 5.08. antiseptics ** Amount 9. purified water 60.010. MAKADEMIAN nuts oil 2.011. cetyl alcohol 2.012. perfume ** Amount (notes 1) bridge-formation mold polyether denaturation silicone: KSG-21 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(notes 2) Bridge formation mold alkyl denaturation silicone compound : KSG-43 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(The manufacture approach)

A: Components 1-4 were mixed to homogeneity.

B: Heating mixing of the components 5-9 was carried out, and it was made homogeneity.

C: Heating mixing of the components 10-12 was carried out.

D: Stirring B, C was added and emulsified and it cooled.

E: D was added and emulsified, stirring A.

The milky lotion obtained as mentioned above was an O/W/O mold milky lotion which mileage feels refreshed lightly, there is neither stickiness nor a feeling of an oil, there is a feeling of transparence, whose makeup **** is good, and change by temperature or the passage of time does not have it, either, and is very excellent also in usability and stability.

[0089]

Example 35: O/W/O mold facial liquid foundation (component) (%)

1. Bridge Formation Mold Polyether Denaturation Silicone (Notes 1) 4.02. Siloxane compound 3 1.03. Decanoic-acid propylene glycol 5.04. Myristic-acid isopropyl 5.05. Pigment 10.06. Yolk origin hydrogenation phospholipid 1.07. glycerol 2. 08.1, 3-butylene glycol 10.09. Antiseptics ** Amount 10. purified water 52.011. Squalane 5.012. Cetyl alcohol 5.013. perfume ** Amount (notes 1) bridge-formation mold polyether denaturation silicone: KSG-21 (Shin-Etsu Chemical [Co., Ltd.] Co., Ltd. make)

(The manufacture approach)

A: Components 1-4 were mixed to homogeneity.

B: Heating mixing of the components 5-10 was carried out, and it was made homogeneity.

C: Heating mixing of the components 11-13 was carried out.

D: Stirring B, C was added and emulsified and it cooled.

E: D was added and emulsified, stirring A.

The facial liquid foundation obtained as mentioned above was O/W/O mold facial liquid foundation which mileage feels refreshed lightly, there is neither stickiness nor a feeling of an oil, there is a feeling of transparence, whose makeup **** is good, and change by temperature or the passage of time does not have it, either, and is very excellent also in usability and stability.

[0090]

[Effect of the Invention] The cosmetics of this invention have the stability which is very good, and they have neither temperature nor change depended with time, and was very excellent in them, when it has light mileage breadth. [of the clean feeling of use which there is no oiliness, and does not try not to carry out gently but is spread and makeup ****] Moreover, when it blends with a skin washing constituent, concordance with others, cosmetics, and sebum dirt is also good, and the cosmetics which have the very good dirt omission effectiveness can also be offered. [descriptions /, such as the above-mentioned feeling of use, usability, and stability with the passage of time,]

[Translation done.]

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1. This document has been translated by computer. So the translation may not reflect the original precisely.

2. **** shows the word which can not be translated.

3. In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

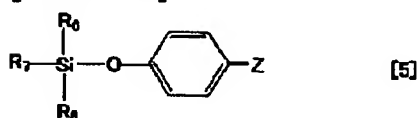
[Field of the Invention] This invention relates to the alkoxysilane which has in more detail the catechol radical protected by the organic substituent about a new organic silicon compound.

[0002]

[Description of the Prior Art] The organic silicon compound from which the phenolic hydroxyl group was protected by the organic substituent and which is shown in the following type [5] and [5'] is known.

[0003]

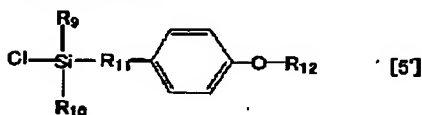
[Formula 5]



[0004] (R6, R7, and R8 are the alkyl groups of carbon numbers 1–6 among a formula, and Z is a vinyl group or a halogen.)

[0005]

[Formula 6]



[0006] (R9, R10, R11, and R12 are the alkyl groups of carbon numbers 1–6 among a formula.) However, the organic silicon compound from which a silicon functional group like an alkoxy silyl radical combined with the end carbon atom of the organic radical which has a catechol radical, and the hydrogen atom of a catechol radical was protected by the organic substituent is not known.

[0007]

[Problem(s) to be Solved by the Invention] The technical problem of this invention offers the alkoxysilane which has the catechol radical protected by the organic substituent, and is to ** in the field of organic synthesis and others by that in fields, such as a new synthetic approach, manufacture of new resin and the ingredient surface treatment approach, and resin reforming.

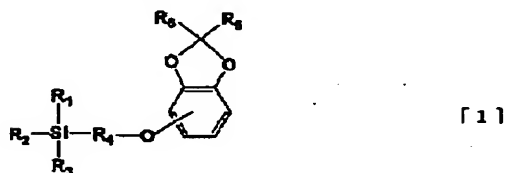
[0008]

[Means for Solving the Problem] this invention person came to complete this invention for such an organic silicon compound being useful as a coupling agent of middle raw materials, such as

organic synthesis, or various ingredients, since it has the carbon functional group which, on the other hand, has one silicon functional group, and was protected by the organic substituent on the other hand based on a header and its knowledge. That is, this invention is an organic silicon compound which has the protected catechol radical and is expressed with the following general formula [1].

[0009]

[Formula 7]

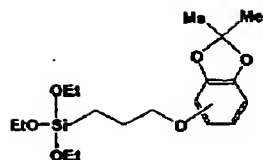


[0010] (You may differ, even if mutually the same, and R1, R2, and R3 are the alkyl groups, phenyl groups, or alkoxy groups of carbon numbers 1-6 among a formula, and at least one of R1, R2, or the R3 is an alkoxy group.) R5 and R6 show the alkyl group or phenyl group of carbon numbers 1-6, and even if mutually the same, they may differ. R4 shows the alkylene group of carbon numbers 2-6.

[0011] Hereafter, this invention is explained in full detail. In the above-mentioned general formula [1] showing the organic silicon compound of this invention, you may differ, even if mutually the same, and R1, R2, and R3 are the alkyl groups, phenyl groups, or alkoxy groups of carbon numbers 1-6, and at least one of R1, R2, or the R3 is an alkoxy group. As an example of the alkyl group of carbon numbers 1-6, and an alkoxy group, there are a methyl group, an ethyl group, a propyl group, an isopropyl group, butyl, an isobutyl radical, a pentyl radical, a hexyl group, a methoxy group, an ethoxy radical, a propyloxy radical, a butyloxy radical, a pentyloxy radical, a hexyloxy radical, etc., and it does not interfere by the shape of a straight chain, and the letter of branching, either. In the above-mentioned general formula [1], R4 may be the alkylene group of carbon numbers 2-6, and any of the shape of a straight chain and the letter of branching are sufficient as it, and it has a dimethylene radical, a trimethylene radical, a tetramethylen radical, a pentamethylene radical, a hexamethylene radical, 2-methyl trimethylene radical, 3-methyl trimethylene radical, etc. as an example. In the above-mentioned general formula [1], R5 and R6 are the alkyl groups or phenyl groups of carbon numbers 1-6, and they have a methyl group, an ethyl group, a propyl group, butyl, a pentyl radical, a hexyl group, and a phenyl group as a desirable example. Any of the shape of a straight chain and the letter of branching are sufficient as R5 and R6, and even if annular, they do not interfere. Since a raw material tends to obtain a desirable thing and composition of the compound of this invention is easy in R1-R6, about R1, R2, and R3, it is an ethoxy radical, and about R4, a carbon number is the straight chain hydrocarbon of 3, and it is a methyl group about R5 and R6. As an alkoxy silyl radical's existence location, Any of the ortho position or the meta position are sufficient to the catechol radical protected by the organic substituent. Although some examples of the compound of such this invention are illustrated, when the above-mentioned thing is taken into consideration, the most desirable thing is a compound expressed with following the [2] type.

[0012]

[Formula 8]



[2]

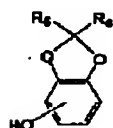
[0013] (Me is a methyl group among a formula and Et is an ethyl group.)

[0014] The compound of this invention can be easily manufactured for example, by following reaction process (1) – (3).

Process (1): Obtain the compound [3] which the trihydroxy benzene and the compound for protection of a catechol radical which have at least two hydroxyl groups in the ortho position are made to react, and is expressed with the following formula [3].

[0015]

[Formula 9]



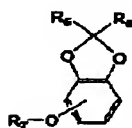
[3]

[0016] (R5 and R6 are [be / it / under / formula / setting] synonymous with the semantics in the above-mentioned general formula [1].)

[0017] Process (2): Obtain the compound [4] which the above-mentioned compound [3] and a halogenation alkene (however, it has the same carbon frame as R4 in the above-mentioned general formula [1], and has a halogen at the molecule end of another side except having a carbon double bond at one molecule end.) are made to react, and is expressed with the following formula [4].

[0018]

[Formula 10]



[4]

[0019] (Among a formula, R7 is the residue except the halogen in a halogenation alkene, and has a carbon double bond at the end.)

[0020] Process (3): Carry out the HIDOROSHI relation reaction of the above-mentioned compound [4] and the silane compound R1R2R3SiH (however, R1, R2, and R3 are synonymous with the semantics in the above-mentioned general formula [1].), and obtain the compound of this invention.

[0021] It explains in more detail about each reaction process of (1) – (3) below.

The trihydroxy benzene in a [process (1)] process (1) has at least two hydroxyl groups in the ortho position, and has pyrogallol, 1 and 2, and 4-trihydroxy benzene as a desirable example. The compound for protection of a catechol radical is a compound which has a reactant radical to R5, R6, and a catechol radical in a molecule (however, semantics and homonymy.). [in / in R5 and R6 / the above-mentioned general formula [1]] It is below the same. . When R5 and R6 are alkyl groups, the desirable reactant radical to a catechol radical is an alkoxy group, and a more desirable reactant radical is a methoxy group. When R5 or R6 is a phenyl group, the desirable

reactant radical to a catechol radical is a halogen, and chlorine more preferably. The following compounds are desirable as a compound for protection of a catechol radical, and it is [0022].

[Formula 11]



[0023]

[Formula 12]

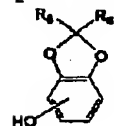


[0024] As the example, there are 2 and 2-dimethoxy butane, 3, and 3-dimethoxy pentane, 5, and 5-dimethoxy nonane, diphenyl dichloromethane, etc.

[0025] The reaction of a process (1) adds a solvent to trihydroxy benzene, and is performed by dropping the compound for protection of a catechol radical under heating reflux. Although this reaction is an equimolar reaction of trihydroxy benzene and the compound for protection of a catechol radical, in order to **** in part out of the system of reaction with the alcohol which carries out a byproduction, it reacts by usually supplying the latter superfluously. The alcohol or hydrogen halide which carries out a byproduction within the system of reaction is made to flow out of the system of reaction, and a reaction is completed. The hydrocarbons like n pentane, n-hexane, a cyclohexane, the petroleum ether, toluene, a xylene, a gasoline, and a ligroin as a desirable solvent; there are diethylether, diisopropyl ether, and ether like a tetrahydrofuran. The following compound [3] is obtained by distilling off a solvent and a volatile component under reduced pressure after completing the above-mentioned reaction. The catechol radical is protected by the organic substituent [(R5) (R6) C=] in this compound [3].

[0026]

[Formula 13]



[3]

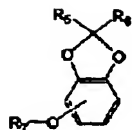
[0027] The halogenation alkene in a [process (2)] process (2) is a compound which has the same carbon frame as R4 in the above-mentioned general formula [1], and has a halogen at the molecule end of another side except having a carbon double bond at one molecule end.

Desirable halogenation alkenes are 2-chloro-1-ethene, a 4-BUROMO-1-butene, 5-chloro-1-pentene, a 6-chloro-1-hexene, etc. The reaction of a process (2) adds a solvent and a base to the compound [3] obtained at the process (1), and is performed by dropping a halogenation alkene under heating reflux.

[0028] Desirable solvents are alcohols like a methanol, ethanol, isopropanol, and a butanol, diethylether, diisopropyl ether, ether like a tetrahydrofuran, water, an acetone, and a polar solvent like dimethylformamide. Desirable bases are a sodium hydroxide, a potassium hydroxide, potassium carbonate, etc. A solvent and a volatile component are distilled off under reduced pressure after completing the reaction of a compound [3] and a halogenation alkene, and the following compound [4] is isolated by vacuum distillation.

[0029]

[Formula 14]



[4]

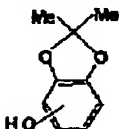
[0030] The reaction of the above-mentioned compound [4] and silane compound ($R_1R_2R_3SiH$) which were obtained at the [process (3)] process (2) is usually performed under existence of a catalyst. As a desirable catalyst, there are a simple substance of the 10th group metal, an organometallic complex, a metal salt, a metallic oxide, etc. from the 8th group, such as cobalt, nickel, a ruthenium, a rhodium, palladium, iridium, and platinum. In these, especially the catalyst of a platinum system is desirable. As a platinum system catalyst, there are chloroplatinic acid 6 hydrate ($H_2PtCl_6 \cdot 6H_2O$), *cis*- $PtCl_2(PhCN)_2$, platinum carbon, a platinum complex ($PtDVTMDS$) that divinyl tetramethyl disiloxane configured. In addition, Ph expresses a phenyl group. The desirable amount of the catalyst used is 0.1 ppm to 1,000 ppm to the amount of a compound [4].

[0031] Moreover, although it is not generally decided in order to depend for the control operation of reaction temperature on the speed of supply of heating from the outside, and a silane compound, a HIDOROSHI relation reaction can be made to usually continue smoothly by holding reaction temperature in the range of room temperature -110 degree C. The compound of this invention is obtained by distilling off a solvent and a volatile component under reduced pressure after reaction termination.

[0032] In the case of a desirable compound [2], it can manufacture as follows among the compounds of this invention. That is, it is made to react, carrying out the demethanol of the trihydroxy benzene and 2,2-dimethoxy propane which have at least two hydroxyl groups in the ortho position, and the compound [5] expressed with the following type [5] is obtained. This compound [5] is a compound which has the catechol radical protected by the organic substituent $[(Me)_2C=]$.

[0033]

[Formula 15]

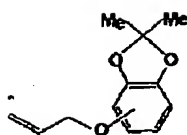


[5]

[0034] A reaction adds a solvent to trihydroxy benzene and is performed by dropping 2,2-dimethoxy propane under heating reflux. The methanol generated within the system of reaction is made to flow out of the system of reaction, and a reaction is completed. A desirable solvent is illustrated in the above-mentioned process (1). The above-mentioned compound [5] is obtained by distilling off a solvent and a volatile component under reduced pressure after completing the reaction of trihydroxy benzene and 2,2-dimethoxy propane. The compound [6] expressed with the following formula [6] is obtained by making a compound [5] react with allyl halide under base existence continuously.

[0035]

[Formula 16]



[6]

[0036] The reaction of a compound [5] and allyl halide adds a solvent and a base to a compound [5], and is performed by dropping allyl halide under heating reflux. A desirable solvent and a desirable base are illustrated at the above-mentioned process (2). Desirable allyl halides are an allyl chloride, an allyl bromide, an allyl iodide, etc. A solvent and a volatile component are distilled off under reduced pressure after completing the reaction of a compound [5] and allyl halide, and a compound [6] is isolated by vacuum distillation.

[0037] Thus, the HIDOROSHI relation reaction of the obtained compound [6] is carried out with triethoxysilane, and the compound [2] of this invention is obtained.

[0038]

[Effect of the Invention] The new alkoxy silane which has the catechol radical protected by the organic substituent by this invention is offered. Since the alkoxy group of the hydrolysis nature combined with the silicon atom exists, the new organic silicon compound of this invention can form siloxane association by the reaction with other organic silicon compounds (a polymer is included), or it can be made it to carry out a coupling reaction to the silanol group in an inorganic compound. Moreover, since 3 functionality alkoxy silane can use the crosslinking reaction of (SiOSi), it is useful as raw materials for manufacture, such as silicone resin or silsesquioxane. On the other hand, the protected catechol radical in the compound of this invention is easily desorbed from a protective group by hydrolysis under acid conditions, turns into a free catechol radical, and functions as a carbon functionality radical or an alkali water solubility radical. by the free catechol radical, the compound of this invention will have two hydroxyls per molecule, alkali water solubility is markedly boiled as compared with the conventional compound from which the phenolic group was protected, and there is the description of being large. Moreover, the component from which it is desorbed in this case is a ketone which is usually rich in volatility, and can be vaporized easily. This property is advantageous to the purpose which lessens non-purity as much as possible in manufacture of the synthetic reaction which uses the compound of this invention, or resin. Since a free catechol radical forms a polar functional group and strong hydrogen bond, the organosilicon compound of this invention can be used also for organic - inorganic hybrid material using hydrogen bond. Since it functions as a double reactivity silicon compound in which the compound of this invention has silicon functionality and the protected carbon functionality as above-mentioned, it is useful as the middle raw material of organic synthesis, the synthetic powder of polymer resin, the modifier of a polymer, and a finishing agent of an inorganic compound.

[0039]

[Example] Hereafter, the example of reference and an example explain this invention concretely.

[0040] The reactor equipped with example of reference 1 agitator, the fractionating tower, and the cooling pipe is made into the bottom of desiccation nitrogen-gas-atmosphere mind, pyrogallol 100.0g (793mmol) and toluene 300mL were taught, and heating reflux was carried out. When 2,2-dimethoxy propane 82.6g (793 mmol) was made dropped gradually, the methanol flowed out with the column top temperature of about 60 degrees C. When the outflow stopped, 2,2-dimethoxy propane 82.6g (793 mmol) of tales doses was made dropped, and it carried out

after dropping termination for heating reflux 3 hours. The brown viscous liquid was obtained by checking that pyrogallol has disappeared out of the system of reaction using a gas chromatography, and distilling off a solvent under reduced pressure. This viscous liquid was melted to ethanol 500mL, and it taught the two-lot flask equipped with reflux tubing. 66.7g (1190 mmol) of sodium hydroxides was taught continuously, and heating reflux was carried out. The system of reaction was made to trickle gradually 144g (1190 mmol) of allyl bromides, and they were performed to it after dropping termination for heating reflux 3 hours. Toluene 200mL was added for ethanol after distilling off under reduced pressure. 110–112 degrees C of boiling points and 86.0g (53 %) of 707Pa transparent and colorless liquids were obtained for the insoluble salt by vacuum distillation the back according to ** in toluene. When 270MHz ¹H-NMR was measured about this liquid, the spectrum of Fig. 1 was obtained. delta value and its attribution are as in the 1st table, and it has checked that the liquid obtained above by this identification result was the following compound [7] containing the catechol protected by the organic substituent.

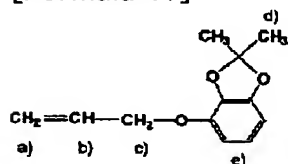
[0041]

[Table 1]

測定法	δ (ppm)	帰属
NMR	1.7	d)
	4.6	e)
	5.3	a)
	6.1	b)
	6.3 ~ 6.7	c)

[0042]

[Formula 17]



[7]

[0043] The reactor equipped with example 1 agitator, the thermometer, and the cooling pipe was made into the bottom of desiccation nitrogen-gas-atmosphere mind, and compound [7] 2.7g (13.1mmol) and triethoxysilane 3.23g (19.7mmol) compounded in the example 1 of reference were taught, and it heated with the oil bath, making the inside of a system stir. In the place where whenever [internal temperature] amounted to 90 degrees C, the xylene solution (1microl, 0.0001mmol) of PtDVTMDS which is a platinum catalyst was added. Stirring neglect was carried out at 90 degrees C after catalyst dropping for 1 hour. Then, under reduced pressure, the volatile component was distilled off and 4.37g (90%) of transparent and colorless liquids was obtained. When 270MHz ¹H-NMR was measured about this liquid, the spectrum of Fig. 2 was obtained. delta value and its attribution were as in the 2nd table. It has checked that the liquid obtained by this was the following compound [8] containing the catechol protected by the organic substituent.

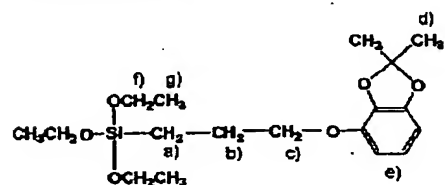
[0044]

[Table 2]

測定法	δ (ppm)	帰属
NMR	0.7	a)
	1.2	g)
	1.7	d)
	1.9	b)
	3.8	f)
	4.1	c)
	6.3~6.8	e)

[0045]

[Formula 18]



[8]

[Translation done.]

*** NOTICES ***

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1. This document has been translated by computer. So the translation may not reflect the original precisely.

2.*** shows the word which can not be translated.

3. In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] Fig. 1 shows ¹H-NMR spectrum of the product obtained in the example 1 of reference.

[Drawing 2] Fig. 2 shows ¹H-NMR spectrum of the product obtained in the example 1.

[Translation done.]

*** NOTICES ***

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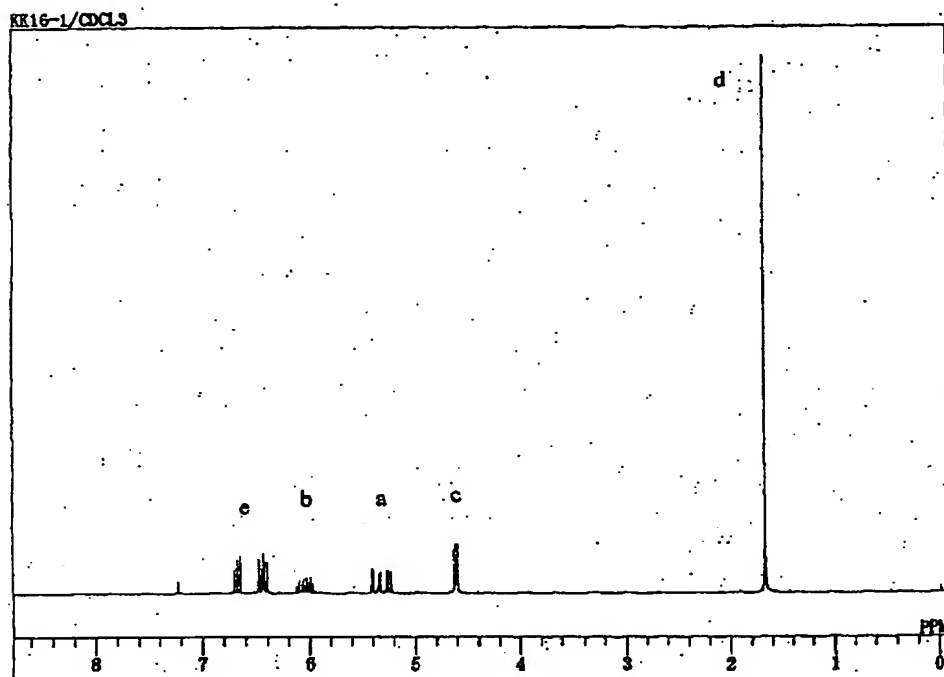
1. This document has been translated by computer. So the translation may not reflect the original precisely.

2.*** shows the word which can not be translated.

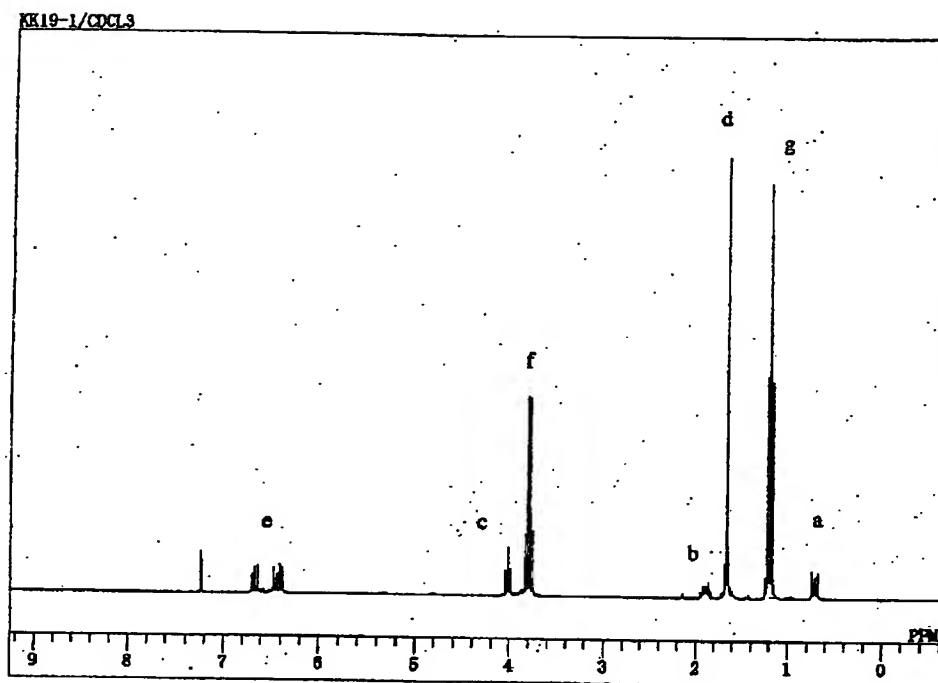
3. In the drawings, any words are not translated.

DRAWINGS

[Drawing 1]



[Drawing 2]



[Translation done.]

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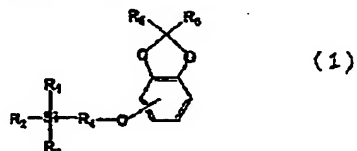
F ターム (参考) 4H049 VN01 VP01 VQ66 VR21 VR43
VS21 VU16 VU21 VU36 VW02
4J035 CA071

(54) 【発明の名称】 保護されたカテコール基を有する有機ケイ素化合物及びその製造方法

(57) 【要約】 (修正有)

【課題】 有機合成その他で新規な合成方法、新規樹脂の製造、材料表面処理方法および樹脂改質等の分野に資するため、有機置換基により保護されたカテコール基を有するアルコキシシランを提供する。

【解決手段】 保護されたカテコール基を有する一般式 1 の有機ケイ素化合物。

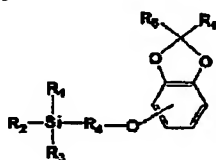


($R_1 \sim R_3$ は C 1 ~ 6 のアルキル基、フェニル基またはアルコキシ基であり、互いに同じでも異なっても良く、 $R_1 \sim R_3$ の 1 つ以上はアルコキシ基である。 R_4 と R_5 は C 1 ~ 6 のアルキル基またはフェニル基を示し、互いに同じでも異なっても良い。 R_4 は C 2 ~ 6 のアルキレン基を示す)

【特許請求の範囲】

【請求項1】保護されたカテコール基を有し、下記一般式〔1〕で表される有機ケイ素化合物。

【化1】

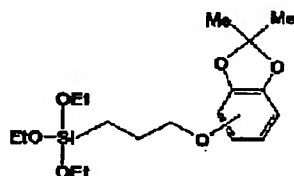


〔1〕

(式中、R₁、R₂およびR₃は炭素数1から6のアルキル基、フェニル基またはアルコキシ基であり、互いに同じであっても異なっても良く、R₁、R₂またはR₃の少なくとも1つはアルコキシ基である。R₃およびR₄は炭素数1から6のアルキル基またはフェニル基を示し、互いに同じであっても異なっても良い。R₄は炭素数2から6のアルキレン基を示す)

【請求項2】請求項1における一般式〔1〕において、R₁、R₂およびR₃がエトキシ基であり、R₄およびR₅がメチル基であり、R₆が炭素数3の直鎖状のアルキル基である、下記構造式〔2〕で表される請求項1記載の有機ケイ素化合物。

【化2】



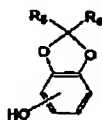
〔2〕

(式中、Meはメチル基であり、Etはエチル基である。)

【請求項3】以下の反応工程(1)～(3)を順次行うことを特徴とする請求項1記載の有機ケイ素化合物の製造方法。

工程(1)：少なくとも2個の水酸基がオルト位にあるトリヒドロキシベンゼンとカテコール基の保護用化合物とを反応させて下記式〔3〕で表される化合物〔3〕を得る。

【化3】

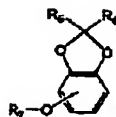


〔3〕

(式中においてR₁およびR₂は上記一般式〔1〕における意味と同義である。)

工程(2)：上記化合物〔3〕とハロゲン化アルケン(但し、一方の分子末端に炭素二重結合を有する以外は上記一般式〔1〕におけるR₄と同じ炭素骨格を有し、他方の分子末端にハロゲンを有する。)とを反応させて下記式〔4〕で表される化合物〔4〕を得る。

【化4】



〔4〕

(式中、R₁は、ハロゲン化アルケンにおけるハロゲンを除いた残基であり、末端に炭素二重結合を有する。)

工程(3)：上記化合物〔4〕とシラン化合物R₄R₅R₆SiH(但し、R₄、R₅およびR₆は上記一般式〔1〕における意味と同義である。)とをヒドロシリレーション反応させる。

【発明の詳細な説明】

【0001】

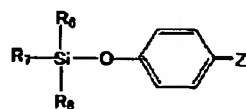
【発明の属する技術分野】本発明は、新規な有機ケイ素化合物に関し、さらに詳しくは、有機置換基により保護されたカテコール基を有するアルコキシシランに関する。

【0002】

【従来の技術】フェノール性水酸基が有機置換基によって保護された、下記式〔5〕、〔5'〕に示す有機ケイ素化合物は知られている。

【0003】

【化5】

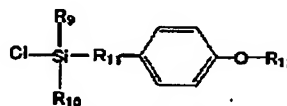


〔5〕

【0004】(式中、R₄、R₅及びR₆は、炭素数1～6のアルキル基であり、Zはビニル基又はハロゲンである。)

【0005】

【化6】



〔5〕

【0006】(式中、R₄、R₅、R₆、R₇、R₈は炭素数1～6のアルキル基である。)

しかし、カテコール基を有する有機基の末端炭素原子にアルコキシシリル基のようなケイ素官能基が結合し、かつ、カテコール基の水素原子が有機置換基で保護された有機ケイ素化合物は知られていない。

【0007】

【発明が解決しようとする課題】本発明の課題は、有機置換基により保護されたカテコール基を有するアルコキシシランを提供し、そのことによって、有機合成その他の分野で新規な合成方法や、新規樹脂の製造、材料表面処理方法および樹脂改質等の分野に資することにある。

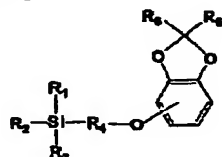
【0008】

【課題を解決するための手段】本発明者は、このような有機ケイ素化合物が、一方では1個のケイ素官能基を有し、他方では有機置換基で保護された炭素官能基を有するために、有機合成などの中間原料や各種材料のカップ

リング剤として有用であることを見出し、その知見に基いて本発明を完成するに至った。即ち、本発明は、保護されたカテコール基を有し、下記一般式〔1〕で表される有機ケイ素化合物である。

【0009】

〔化7〕



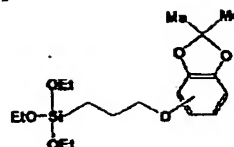
〔1〕

【0010】(式中、 R_1 、 R_2 および R_3 は炭素数1から6のアルキル基、フェニル基またはアルコキシ基であり、互いに同じであっても異なっても良く、 R_1 、 R_2 または R_3 の少なくとも1つはアルコキシ基である。 R_4 および R_5 は炭素数1から6のアルキル基またはフェニル基を示し、互いに同じであっても異なっても良い。 R_4 は炭素数2から6のアルキレン基を示す)

【0011】以下、本発明について詳述する。本発明の有機ケイ素化合物を表す上記一般式〔1〕において、 R_1 、 R_2 および R_3 は炭素数1から6のアルキル基、フェニル基またはアルコキシ基であり、互いに同じであっても異なっても良く、 R_1 、 R_2 または R_3 の少なくとも1つはアルコキシ基である。炭素数1から6のアルキル基及びアルコキシ基の具体例として、メチル基、エチル基、プロピル基、イソプロピル基、ブチル基、イソブチル基、ペンチル基、ヘキシル基、メトキシ基、エトキシ基、プロピルオキシ基、ブチルオキシ基、ペンチルオキシ基、ヘキシルオキシ基等があり、直鎖状でも分岐状でも差し支えない。上記一般式〔1〕において、 R_4 は炭素数2から6のアルキレン基で、直鎖状、分岐状のいずれでもよく、具体例として、ジメチレン基、トリメチレン基、テトラメチレン基、ペンタメチレン基、ヘキサメチレン基、2-メチルトリメチレン基、3-メチルトリメチレン基等がある。上記一般式〔1〕において、 R_5 および R_6 は炭素数1から6のアルキル基またはフェニル基であり、好ましい具体例として、メチル基、エチル基、プロピル基、ブチル基、ペンチル基、ヘキシル基及びフェニル基がある。 R_4 および R_6 は、直鎖状又は分岐状の何れでも良く、また環状であっても差し支えない。 $R_1 \sim R_6$ において好ましいものは、原料が得易く、本発明の化合物の合成が容易なことから、 R_1 、 R_2 および R_3 についてはエトキシ基であり、 R_4 については炭素数が3の直鎖状炭化水素であり、 R_5 および R_6 についてはメチル基である。アルコキシシリル基の存在位置としては、有機置換基により保護されたカテコール基に対して、オルト位又はメタ位のいずれでも構わない。このような本発明の化合物の具体例はいくつか例示されるが、上記のことを考慮すると、もっとも好ましいものは、下記〔2〕式で表される化合物である。

【0012】

〔化8〕



〔2〕

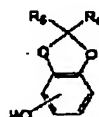
【0013】(式中、Meはメチル基であり、Etはエチル基である。)

10 【0014】本発明の化合物は、例えば以下の反応工程(1)～(3)により容易に製造することができる。

工程(1)：少なくとも2個の水酸基がオルト位にあるトリヒドロキシベンゼンとカテコール基の保護用化合物とを反応させて下記式〔3〕で表される化合物〔3〕を得る。

【0015】

〔化9〕



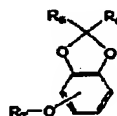
〔3〕

【0016】(式中において R_1 および R_6 は上記一般式〔1〕における意味と同義である。)

【0017】工程(2)：上記化合物〔3〕とハロゲン化アルケン(但し、一方の分子末端に炭素二重結合を有する以外は上記一般式〔1〕における R_4 と同じ炭素骨格を有し、他方の分子末端にハロゲンを有する。)とを反応させて下記式〔4〕で表される化合物〔4〕を得る。

30 【0018】

〔化10〕



〔4〕

【0019】(式中、 R_7 は、ハロゲン化アルケンにおけるハロゲンを除いた残基であり、末端に炭素二重結合を有する。)

40 【0020】工程(3)：上記化合物〔4〕とシラン化合物 $R_1R_2R_3SiH$ (但し、 R_1 、 R_2 および R_3 は上記一般式〔1〕における意味と同義である。)とをヒドロシリレーション反応させて本発明の化合物を得る。

【0021】以下に(1)～(3)の各反応工程について更に詳しく説明する。

【工程(1)】工程(1)におけるトリヒドロキシベンゼンは、少なくとも2個の水酸基がオルト位にあるものであり、好ましい例として、ピロガロール、1,2,4-トリヒドロキシベンゼンがある。カテコール基の保護用化合物は、分子中に、 R_1 、 R_6 及びカテコール基に対する反応性基を有する化合物である(但し、 R_1 、 R_6 は上記一

般式〔1〕における意味と同義。以下同じ。）。 R_4 及び R_5 がアルキル基である場合、カテコール基に対する好ましい反応性基はアルコキシ基であり、より好ましい反応性基はメトキシ基である。 R_4 又は R_5 がフェニル基である場合、カテコール基に対する好ましい反応性基はハロゲンであり、より好ましくは塩素である。カテコール基の保護用化合物として以下の化合物が好ましく、

〔0022〕

〔化11〕



〔0023〕

〔化12〕

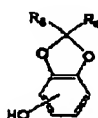


〔0024〕その例として、2,2-ジメトキシブタン、3,3-ジメトキシペンタン、5,5-ジメトキシノナン、ジフェニルジクロロメタン等がある。

〔0025〕工程（1）の反応は、トリヒドロキシベンゼンに溶媒を加え、加熱還流下でカテコール基の保護用化合物を滴下することにより行う。この反応はトリヒドロキシベンゼンとカテコール基の保護用化合物との等モル反応であるが、副生するアルコールと共に反応系外へ一部溜出するため、通常後者を過剰に供給して反応を行う。反応系内で副生するアルコール又はハロゲン化水素を反応系外へ流出させ、反応を完結させる。好ましい溶媒として、*n*-ペンタン、*n*-ヘキサン、シクロヘキサン、石油エーテル、トルエン、キシレン、ガソリン、リグロインのような炭化水素類；ジエチルエーテル、ジイソプロピルエーテル、テトラヒドロフランのようなエーテル類がある。上記の反応が終了後、溶媒および揮発成分を減圧下留去することにより下記化合物〔3〕を得る。この化合物〔3〕において、カテコール基は有機置換基〔 $(R_1)(R_2)C=$ 〕により保護されている。

〔0026〕

〔化13〕



〔3〕

〔0027〕〔工程（2）〕工程（2）におけるハロゲン化アルケンは、一方の分子末端に炭素二重結合を有する以外は上記一般式〔1〕における R_4 と同じ炭素骨格を有し、他方の分子末端にハロゲンを有する化合物である。好ましいハロゲン化アルケンは、2-クロロ-1-エテン、4-ブロモ-1-ブテン、5-クロロ-1-ペンテン、6-クロロ-1-ヘキセン等である。工程（2）の反応は、工程（1）で得た化合物〔3〕に溶媒、塩基を加え、加熱還流下、ハロゲン化アルケンを滴下することにより行う。

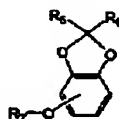
〔0028〕好ましい溶媒は、メタノール、エタノール、イソプロパノール、ブタノールのようなアルコール

10

類、ジエチルエーテル、ジイソプロピルエーテル、テトラヒドロフランのようなエーテル類、水、アセトン、ジメチルホルムアミドのような極性溶媒である。好ましい塩基は、水酸化ナトリウム、水酸化カリウム、炭酸カリウム等である。化合物〔3〕とハロゲン化アルケンとの反応が終了後、溶媒および揮発成分を減圧下留去し、減圧蒸留によって下記化合物〔4〕を単離する。

〔0029〕

〔化14〕



〔4〕

20

〔0030〕〔工程（3）〕工程（2）で得た上記化合物〔4〕とシラン化合物〔 $R_1R_2R_3SiH$ 〕との反応は、通常、触媒の存在下で行う。好ましい触媒として、コバルト、ニッケル、ルテニウム、ロジウム、パラジウム、イリジウム、白金等の第8族から第10族金属の単体、有機金属錯体、金属塩、金属酸化物等がある。これらの中で、白金系の触媒が特に好ましい。白金系触媒としては、塩化白金酸六水和物〔 $H_2PtCl_6 \cdot 6H_2O$ 〕、*cis*- $PtCl_2(PhCN)_2$ 、白金カーボン、ジビニルテトラメチルジシロキサンが配位した白金錯体〔 $Pt(dVTMDS)$ 〕等がある。なお、Phはフェニル基を表わす。触媒の好ましい使用量は、化合物〔4〕の量に対して、0.1ppmから1,000ppmである。

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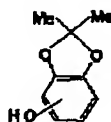
〔0031〕また、反応温度の制御操作は、外部からの加熱およびシラン化合物の供給速度に依存するため、一概に決められないが、通常、反応温度を室温〜110℃の範囲に保持することで、ヒドロシリレーション反応を円滑に継続させることができる。反応終了後、溶媒および揮発成分を減圧下留去することにより本発明の化合物を得る。

〔0032〕本発明の化合物の内、好ましい化合物

〔2〕の場合、次のようにして製造することができる。即ち、少なくとも2個の水酸基がオルト位にあるトリヒドロキシベンゼンと2,2-ジメトキシプロパンを脱メタノールさせながら反応させ、下記式〔5〕で表される化合物〔5〕を得る。この化合物〔5〕は有機置換基〔 $(Me)_2C=$ 〕により保護されたカテコール基を有する化合物である。

〔0033〕

〔化15〕



〔5〕

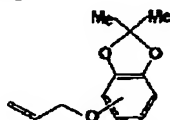
〔0034〕反応は、トリヒドロキシベンゼンに溶媒を加え、加熱還流下で2,2-ジメトキシプロパンを滴下することにより行う。反応系内で生成されるメタノールを反

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応系外へ流出させ、反応を完結させる。好ましい溶媒は、上記工程(1)において例示したものである。トリヒドロキシベンゼンと2,2-ジメトキシプロパンとの反応が終了後、溶媒および揮発成分を減圧下留去することにより上記化合物[5]を得る。続けて化合物[5]を塩基存在下、ハロゲン化アリルと反応させることにより、下記式[6]で表される化合物[6]を得る。

【0035】

【化16】



【6】

【0036】化合物[5]とハロゲン化アリルとの反応は、化合物[5]に溶媒、塩基を加え、加熱還流下、ハロゲン化アリルを滴下することにより行う。好ましい溶媒及び好ましい塩基は、上記工程(2)で例示したものである。好ましいハロゲン化アリルは、塩化アリル、臭化アリル、ヨウ化アリル等である。化合物[5]とハロゲン化アリルとの反応が終了後、溶媒および揮発成分を減圧下留去し、減圧蒸留によって化合物[6]を単離する。

【0037】このようにして得られた化合物[6]を、トリエトキシシランとヒドロシリレーション反応させ、本発明の化合物[2]を得る。

【0038】

【発明の効果】本発明によって、有機置換基により保護されたカテコール基を有する新規なアルコキシシランが提供される。本発明の新規な有機ケイ素化合物は、ケイ素原子に結合した加水分解性のアルコキシ基が存在するため、他の有機ケイ素化合物(ポリマーを含む)との反応によりシロキサン結合を形成したり、無機化合物中のシラノール基とカップリング反応させることができる。また、3官能性アルコキシシランは、(SiOSi)の架橋反応を利用することができるので、シリコーンレジン又はシルセスキオキサン等の製造用原料として有用である。一方、本発明の化合物における保護されたカテコール基は、酸性条件下で加水分解により容易に保護基を脱離して、フリーのカテコール基となり、炭素官能性基またはアルカリ水溶性基として機能する。フリーのカテコール基により、本発明の化合物は、1分子当たり2個のヒドロキシル基を有することになり、フェノール基が保護された従来の化合物に比較して、アルカリ水溶性は格段に大きいという特徴がある。また、この際脱離する成分は通常揮発性に富むケトンであり、容易に揮散させることができる。この特性は、本発明の化合物を用いる合成反応や樹脂の製造において、不純分をできるだけ少なくする目的に有利である。フリーのカテコール基は極性官能基と強い水素結合を形成するので、本発明の有機珪素化合物は、水素結合を利用した有機-無機ハイブリ

ッド材料へも利用できる。上記の通り、本発明の化合物は、ケイ素官能性および保護された炭素官能性をもつ複反応性ケイ素化合物として機能するので、有機合成の中間原料、ポリマー樹脂の合成原料、ポリマーの改質剤、無機化合物の表面処理剤として有用である。

【0039】

【実施例】以下、本発明を参考例および実施例によって具体的に説明する。

【0040】参考例1

- 10 攪拌機、精留塔及び冷却管を備えた反応器を乾燥窒素雰囲気下にして、ピロガロール100.0g(793mmol)、トルエン300mLを仕込み、加熱還流させた。2,2-ジメトキシプロパン82.6g(793mmol)を徐々に滴下させると、塔頂温度約60℃でメタノールが流出した。流出が止まったら、同量の2,2-ジメトキシプロパン82.6g(793mmol)を滴下させ、滴下終了後、加熱還流3時間行った。ガスクロマトグラフィーを用いてピロガロールが反応系内から消失したのを確認し、溶媒を減圧下で留去することにより、褐色粘性液体を得た。この粘性液体をエタノール50
- 20 0mLに溶かし、還流管を備えた2口フラスコに仕込んだ。続けて水酸化ナトリウム66.7g(1190mmol)を仕込み、加熱還流させた。反応系に臭化アリル144g(1190mmol)を徐々に滴下させ、滴下終了後、加熱還流3時間行った。エタノールを減圧下留去後、トルエン200mLを加えた。トルエンに不溶の塩をろ別後、減圧蒸留によって、沸点110~112℃/707Paの無色透明の液体86.0g(53%)を得た。この液体について270MHzの¹H-NMRの測定を行ったところ、第1図のスペクトルを得た。δ値とその帰属は第1表のとおりであり、この同定結果により、
- 30 上記で得られた液体は、有機置換基により保護されたカテコールを含有する下記化合物[7]であることが確認できた。

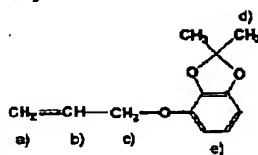
【0041】

【表1】

測定法	δ (ppm)	帰属
NMR	1.7	d)
	4.6	c)
	5.3	a)
	6.1	b)
	6.3~6.7	e)

40 【0042】

【化17】



【7】

【0043】実施例1

攪拌機、温度計及び冷却管を備えた反応器を乾燥窒素雰囲気下にして、参考例1で合成した化合物[7]2.7g(13.1mmol)、トリエトキシシラン3.23g(19.7mmol)

を仕込み、系内を攪拌させながらオイルバスで加熱した。内温度が90℃に達したところで、白金触媒であるPt DVTMSのキシレン溶液(1μl, 0.0001mmol)を加えた。触媒滴下後、1時間、90℃で攪拌放置した。その後、減圧下、揮発成分を留去し無色透明の液体4.37g(90%)を得た。この液体について270MHzの¹H-NMRの測定を行ったところ、第2図のスペクトルを得た。δ値とその帰属は第2表のとおりであった。これにより得られた液体は、有機置換基により保護されたカテコールを含有する下記化合物〔8〕であることが確認できた。

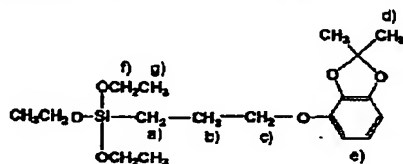
*〔0044〕

〔表2〕

測定法	δ (ppm)	帰属
NMR	0.7	a)
	1.2	g)
	1.7	d)
	1.9	b)
	3.8	f)
	4.1	c)
	6.3~6.8	e)

〔0045〕

*10 〔化18〕



〔8〕

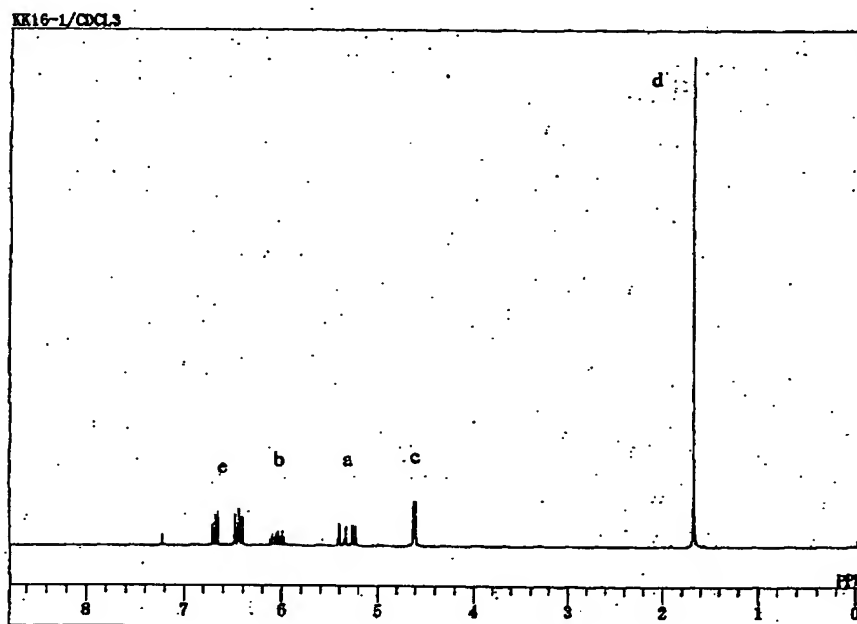
〔図面の簡単な説明〕

〔図1〕第1図は参考例1で得られた生成物の¹H-NMRスペクトルを示す。

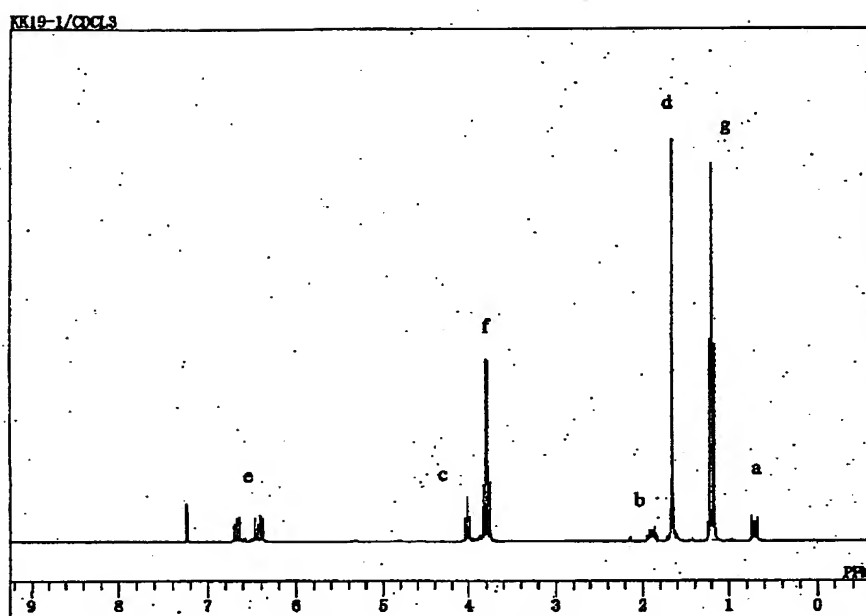
※〔図2〕第2図は実施例1で得られた生成物の¹H-NMRスペクトルを示す。

※

〔図1〕



【図2】



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